TRIAL BURN PLAN

MONSANTO CHEMICAL COMPANY

QUEENY PLANT

CAC INCINERATOR

Prepared by:

James A. Peters

Terran Corporation

May 1988

110

Monsanto

Monsanto Chemical Co.
1700 South Second Street
St. Louis, Missouri 63177
Phone: (314) 622-1400

June 9, 1988

Mr. Thomas C. Pauling
Environmental Engineer
Waste Management Program
Division of Environmental Quality
Missouri Department of Natural Resources
P.O. Box 176
Jefferson City, MO 65102

Dear Mr. Pauling:

Enclosed are three copies of the revised Trial Burn Plan for the Monsanto Queeny Plant CAC incinerator. Two copies have been sent to Mr. John Smith, USEPA Region VII. Page 34 provides the proposed timetable for completing the Trial Burn following plan approval. We look forward to receiving such.

Please contact me if you have any questions.

Sincerely,

Robert F. Boland

Robert F. Boland, PE
Environmental Protection
Superintendent

cc: Mr. John Smith
U. S. Environmental Protection Agency
Region VII
726 Minnesota Avenue
Kansas City, KS 66101
with 2 enclosures

TABLE OF CONTENTS

Section		Page
I	Introduction	1
II	Reporting Format	1
III	Wastes to be Burned	9
IV	Detailed Engineering Description of the Incinerator	13
V	Trial Burn Protocol	27
VI	Sampling and Analysis Plan	35
VII	Quality Assurance/Quality Control Plan	59
	Appendix A -- Engineering Drawings	
	Appendix B -- Spare Parts Lists	
	Appendix C -- Incinerator Operating Procedures	
	Appendix D -- Nonstandard Sampling and Analysis Methods	
	Appendix E -- Field and Laboratory Data Sheets and Forms	

LIST OF FIGURES

Figure No.		Page
IV-1	Schematic diagram of the Monsanto-Queeny plant CAC incinerator system.	14
IV-2	Location of CAC incinerator process control sensors and monitors.	20
IV-3	Schematic diagram of instrumentation for CAC oxidizer section.	22
VI-1	Schematic diagram of sampling locations.	36
VI-2	Diagram of the CAC residue sampling location.	37
VI-3	Schematic of exhaust gas sampling train for particulate and chloride.	45
VI-4	Schematic of volatile organic sampling train (VOST).	49
VI-5	Sorbent trap configurations.	50
VI-6	Schematic of typical VOST sorbent trap desorption and purge and trap apparatus.	56
VI-7	CEM sampling system for CO and O_2.	58
VII-1	Organization of PEI corporate quality assurance.	62
VII-2	Emission measurement group organization chart.	64
VII-3	Laboratory organization chart.	65
VII-4	Project responsibilities and quality assurance loops.	66
VII-5	Three part sample label.	68

LIST OF TABLES

Table No.		Page
II-1	Trial Burn Report Outline -- Main Report	2
II-2	Trial Burn Report Outline -- Appended Information	5
II-3	Example Summary of Results -- Process Operation - Test Condition No. 1	6
II-4	Example Summary of Results -- Emission Performance - Test Condition No. 1	7
II-5	Example Average Trial Burn Results at Test Conditions	8
III-1	CAC Residue Characteristics -- Physical/Chemical	10
III-2	Summary of Incinerated Waste Streams, CAC Incinerator, Monsanto-Queeny Plant	11
IV-1	Summary Design Information	15
IV-2	Summary of CAC Incinerator Process Monitors	18
IV-3	Summary of CAC Incinerator Instrument Set Points and Function	24
V-1	POHC Selection Criteria Summary	30
V-2	Summary of Test Condition Parameter Targets for CAC Incinerator System	33
V-3	Schedule for CAC Incinerator Trial Burn Completion	34
V-4	Summary of Waste Quantities to be Burned During Monsanto-Queeny Plant CAC Incinerator Trial Burn Performance Testing	34
VI-1	Summary of Sampling Methods, CAC Incinerator, Monsanto-Queeny Plant, St. Louis, Missouri	38
VI-2	Summary of Analysis Methods, CAC Incinerator, Monsanto-Queeny Plant, St. Louis, Missouri	39

LIST OF TABLES

Table No.		Page
VI-3	Example Calculation of Gas Sample Size Needed to Demonstrate 99.999% DRE	48
VII-1	Quality Assurance Measures for the Trial Burn	60
VII-2	Quality Assurance Objectives for CAC Incinerator Trial Burn Measurement Data	63
VII-3	Field Equipment Calibration Summary	70
VII-4	Field Checks of Sampling Equipment	71

TRIAL BURN PLAN

I. INTRODUCTION

The CAC incinerator is located at the southeast corner of the Monsanto Chemical Company's John F. Queeny plant in the city of St. Louis, Missouri. The incinerator is used to treat liquid wastes generated from the CAC and Alachlor processes during the manufacture of a major agricultural herbicide.

The incinerator system consists of a forced draft, liquid injection burner/thermal oxidizer chamber, quench pot, and an air pollution control scrubber. Liquid wastes are fed to the gas-fired incinerator at a maximum rate of 1400 pounds per hour. The heat capacity of the CAC incinerator is 17 million Btu per hour (mmBtu/hr).

The purpose of this trial burn plan is to include a special submittal as part of the RCRA permit application describing how the incinerator system will be tested to satisfy the performance requirements specified by the regulations and to establish the operating conditions for the incinerator to assure that performance standards will be met.

II. REPORTING FORMAT

One purpose of the trial burn performance test is to provide the EPA with the data needed to properly assess the incineration system performance and enable a route to set permit conditions that will allow the incinerator to operate in compliance thereafter. This section provides a description of what the final report will look like, so that a) the test conditions chosen are more easily understood and b) EPA can decide if the proposed data package will be adequate.

Table II-1 presents the proposed trial burn results final report outline and format for the main report. From sections in the main report, references to the raw data in appendices will be made where appropriate. Table II-2 presents the proposed outline of the trial burn results appended sections.

The content of tabular information is important to the ease of understanding the test results and the relationship to process parameters. Table II-3 presents the example summary of results format for process operation parameters to be measured/monitored during the trial burn. Table II-4 presents the summary of results format for emission performance. Table II-5 presents the format for displaying the average trial burn results at the test conditions.

TABLE II-1. TRIAL BURN REPORT OUTLINE -- MAIN REPORT

Report organization	Specific information
Title page	Report title Facility name/location, Dates Name of company performing testing Author(s) Report approvals
Table of contents/list of tables and figures	
1.0 Summary of Test Results	
* Process operation	Test dates Residence times Combustion temperatures Heat input (firing) rate Waste feed rates Auxiliary fuel feed rates Summary of quench parameters Summary of APCE parameters Stack height Stack exit velocity Stack temperature Stack excess O_2 Stack CO concentrations
* Emissions performance	Test dates DREs Particulate emissions HCl emissions HCl control efficiency Stack gas flow rates O_2 concentrations CO_2 concentrations CO concentrations
2.0 Introduction	
* Background	Brief discussion of incinerator type Design data summary Objectives for trial burn Planned test matrix and deviations Description of wastes/fuels
* Non-standard	Description of any unusual test methodologies Discussion of any special problems encountered

TABLE II-1. TRIAL BURN REPORT OUTLINE -- MAIN REPORT (Continued)

Report organization		Specific information
3.0	Performance Results	
3.1	POHCs	Input rates Emission rates DREs
3.2	Chlorides	Input rates Emission rates Removal efficiencies
3.3	Particulates	Concentrations
4.0	Process Operating Conditions	
4.1	Process Overview	Brief description Process diagram Summary of process monitors
4.2	Incinerator Operating Conditions	
	* Combustion temperatures	Burner plenum temperature Oxidizer temperature
	* Waste feed/aux fuel data	Brief description/firing locations
	* Waste burners	CAC atomization/burner pressures
	* Air flow data	Flow rates/velocities from EPA 5 Blower data Draft measurements
	* Residue generation	Quench water flow rates Scrubber water flow rates
4.3	APCE Operating Conditions	
	* Quench	Inlet temperature Exit temperature Water flow rate Inlet/exit pH Liquid levels
	* Packed tower scrubber	Inlet temperature Water flow rate Exit temperature Inlet/exit pH Liquid levels
5.0	Sampling and Analysis Results	
5.1	Methods Description	Summary table Diagram indicating sampling locations

TABLE II-1. TRIAL BURN REPORT OUTLINE -- MAIN REPORT (Continued)

Report organization		Specific information
5.2	Waste Feed and Fuel Characteristics	
	* Physical characteristics	Moisture
		Ash
		Btu value
		Viscosity
		Specific gravity
	* Chemical characteristics	Elemental analyses
		Chlorine content
		POHCs
		Other App. VIII compounds
		Metals
5.3	Stack Gas Concentration Data	
	* Gases	
	- POHCs	Volatiles and semivolatiles
	- CEMS	CO
		O_2
	- Orsat	CO_2
		O_2
	- Other	Particulates
		HCl
		Moisture
5.4	APCE Aqueous Streams	POHCs
		Chlorides
		TDS
		TSS
		pH
		TOX
5.5	QA/QC Results	Data quality objectives
		Comparison of QC results vs. goals
		Holding times
		QA/QC documentation discussion

TABLE II-2. TRIAL BURN REPORTING FORMAT -- APPENDED INFORMATION

Appendix format	Content
Emission Results	Computer printouts Example calculations
Detailed S&A Results	
* POHCs	Concentration in each sample Sampling periods Trip and blank values Averages
* Chloride	Concentration Impinger volumes Blank values
* Particulate	Nozzle/probe wash weights Filter weights
* Analytical results	Laboratory reports/data
Raw Data Logs	
* Stack sampling data	Field data forms
* CEM data	Strip charts/printouts
* Process sample data	Field data forms
* Process data	Log sheets, strip charts, computer printouts
Sample Traceability Records	Chain-of-custody forms, analysis logs
QA/QC Results	Surrogate recoveries Replicate analyses, percent differences Blind audit samples Calibration data Performance audit(s) results Instrument detection limits Method blank summaries
S&A Methods	Summary of standard methods Nonstandard methods or modifications
Chromatograms	Waste analysis examples Emission analysis examples
Project Participants	List of names, titles, affiliations

TABLE II-3. EXAMPLE SUMMARY OF RESULTS -- PROCESS OPERATION[a] - TEST CONDITION NO. 1

Parameter	Units	Run No. 1-1	1-2	1-3
Test date	--			
Combustion gas flow rate	acfm dscfm			
Combustion air flow rate	acfm			
Heat input rate	Btu/hr			
Oxidizer temperature[b]	°C			
Oxidizer chamber pressure	psig			
Quench outlet temperature[c]	°C			
Scrubber outlet temperature	°C			
Quench water flow rate	gpm			
Scrubber water flow rate	gpm			
Quench/scrubber inlet	pH			
Quench outlet	pH			
Scrubber outlet	pH			
Stack height	ft			
Stack exit velocity	fps			

TABLE II-3. EXAMPLE SUMMARY OF RESULTS -- PROCESS OPERATION[a] - TEST CONDITION NO. 1 (Continued)

Parameter	Units	Run No. 1-1	1-2	1-3
Stack temperature	°C			
Stack excess O_2[d]	%			

[a] Average of readings taken during each run.
[b] Approximate quench inlet temperature.
[c] Approximate scrubber inlet temperature.
[d] Orsat analysis.

TABLE II-4. EXAMPLE SUMMARY OF RESULTS -- EMISSION PERFORMANCE - TEST CONDITION NO. 1

Parameter	Units	Run No. 1-1	1-2	1-3	Average Std dev CV[a]
Test date	--				
DRE - Ethylene dichloride	%				
DRE - Tetrachloro-ethylene	%				
Particulates[b]	mg/m^3				
HCl emissions	lb/hr				
HCl removal effic.	%				
Stack gas flow rate	dscfm				
	acfm				
Oxygen	%				
Carbon monoxide[c]	ppm				

[a] CV = Coefficient of variation.
[b] Corrected to 7% O_2.
[c] Uncorrected.

TABLE II-5. EXAMPLE AVERAGE TRIAL BURN RESULTS AT TEST CONDITIONS

Test condition	Permit target	1	2	3	4
Oxidizer temperature (^{o}C)	TBD				
Combustion air flow rate (acfm)	TBD				
Stack exit velocity (fps)	TBD				
DRE - Ethylene dichloride (%)	99.99				
DRE - Tetrachloro-ethylene (%)	99.99				
Particulate concentration (mg/m^3)	180				
HCl control effic. (%)	99.0				
CO concentration (ppm)	100				
O_2 concentration (%)	TBD				
Feed rates: (lb/hr)					
CAC residue	TBD				
Total inorganic ash	TBD				
Total chlorine	TBD				
Heat input (Btu/hr)	TBD				
Atomization pressure (psig)	TBD				
Quench water flow rate (gpm)	TBD				
Scrubber water flow rate (gpm)	TBD				

[a] TBD = To be determined from trial burn data.

III. WASTES TO BE BURNED

The first task in selecting trial burn test conditions involves review of the individual waste streams and burn rate requirements. The CAC incinerator facility is piped to incinerate two liquid waste streams -- CAC waste which is a hazardous waste and Azo residue which is a nonhazardous waste. Both waste streams are burned separately in the CAC incinerator.

CAC residue results from impurities formed in the CAC Intermediate process chlorinator, which are separated in a light ends column and solvent column by distillation. The light ends column feeds overhead light ends to the CAC residue storage tank while the solvent column feeds bottoms to the CAC residue tank. Both feed on a continuous basis as the process is operating.

Tables III-1 and III-3 summarize the major physical characteristics and composition of the CAC waste stream.

III.1 Chemical and Physical Analyses

The analysis results of Table III-1 came from a June/July 1984 characterization of CAC residue by Monsanto Agricultural Research in St. Louis, Missouri, and from a April 1988 sample characterized by PEI Associates, Inc. of Cincinnati, Ohio. The MAC organic analysis was performed using a modified MAC Research Method No. 130, which is the corporate standard procedure for determination of 1,2-dichloroethane in CAC waste. This method is included in Appendix D. The principle of the method is that the internal standard toluene is added to a volumetric flask containing an accurately weighed sample of CAC waste and diluted with dichloromethane to volume. The dichloromethane solution is injected into a capillary gas chromatograph equipped with a 60 m X 0.25 mm ID DB-5 fused silica open tubular column and a flame ionization detector. Quantitation is based on the ratio of peak areas, analyte:toluene, as measured by an electronic integrator. Four samples, each one week apart, from the CAC process were analyzed as well as a single sample from the solvent bottoms fraction. These analyses established the ranges, shown in coefficient of variation (CV) in table III-1. The 1988 analysis results came from use of EPA Methods 3580 (dilution in methanol) and 8240 (GC/MS for volatiles) for the organic constituents.

Table III-2 presents the summary of hazardous waste streams which are incinerated in the facility, EPA waste I.D. and type, and maximum feed rate to the incinerator.

TABLE III-1. CAC RESIDUE CHARACTERISTICS -- PHYSICAL/CHEMICAL

Parameter	CAC residue	CAC solvent bottoms only
Physical		
Btu content[a]	8260	7290
Viscosity[a]	1.56 cS +/- 19%[b]	7.31 cS
Specific gravity[c]	1.41	NA
Chemical		
Elemental[a]		
Carbon	34.5% +/- 3%	38.1%
Hydrogen	3.9% +/- 3%	4.2%
Oxygen (by difference)	23.2%	25.4%
Chlorine	38.4% +/- 4%	32.3%
Metals[c]		
Arsenic	<0.20 ppm	NA
Cadmium	<0.07 ppm	NA
Chromium	0.27 ppm	NA
Lead	<6.2 ppm	NA
Organic constituents[a]		
Acetyl chloride** (CAS# 75-36-5)	34.5% +/- 19%	<1%
Propargyl chloride	0.5% +/- 40%	<0.1%
Butyrolacetone	2.4% +/- 29%	30.5%
2-Chloroethyl-4-chlorobutyrate	7.1% +/- 23%	18.4%
Acetic acid	2.0% +/- 45%	<0.5%
1,2-Dichloroethane**[c]	0.24% +/- 17%	<0.5%
Acetic anhydride	3.3% +/- 9%	<0.5%
Chloroacetyl chloride	11.1% +/- 11%	1.4%
Chloromethane	88 ppm +/- 31%	<25 ppm
Dichloromethane** (CAS# 75-09-2)	35 ppm +/- 43%	<25 ppm
Chloroform** (CAS# 67-66-3)	127 ppm +/- 24%	137 ppm
Carbon tetrachloride** (CAS# 56-23-5)	67 ppm +/- 46%	<25 ppm
Tetrachloroethane** (CAS# 630-20-6 or 79-34-5)	37 ppm +/- 19%	<25 ppm

TABLE III-1. CAC RESIDUE CHARACTERISTICS -- PHYSICAL/CHEMICAL (Continued)

Parameter	CAC residue	CAC solvent bottoms only
2,3-Dichloro-1-propene** (CAS# 26952-23-8)	442 ppm +/- 52%	664 ppm
1,2,3-Trichloropropene	500 ppm +/- 72%	431 ppm
Closure[d]		
High boiling tars	18.5%	approx 30%
Dichloroacetic anhydride	18.8%	approx 7%

** Appendix VIII compound.
[a] From Monsanto Agricultural Company Research analyses of four samples taken one week apart.
[b] When presented with (+/-), is average of four analyses plus coefficient of variation (in %).
[c] From PEI Associates,Inc. analysis of 4/88 sample.
[d] From other CAC waste characterizations.

TABLE III-2. SUMMARY OF INCINERATED WASTE STREAMS, CAC INCINERATOR, MONSANTO -QUEENY PLANT

Waste name	EPA waste ID code	Waste description	RCRA App. VIII compounds	Disposal requirements (lb/hr)
CAC residue	D002 & D003	Liquid organic waste	Ethylene dichloride Carbon tetra-chloride Tetrachloro-ethane, N.O.S. Chloroform Dichloromethane Dichloropropene, N.O.S. Acetyl chloride	800 to 1400

III.2 Procedures to Prevent Reactions

Regulations given in 40 CFR 270.14(b)(9) require a description of precautions to prevent accidental reaction of reactive or incompatible wastes as required to demonstrate compliance with Part 264.17. The CAC residue is classified as a reactive waste (D003) because, when mixed with water, it generates toxic gases, vapors or fumes (HCl) in a quantity sufficient to present a danger [261.23(a)(4)]. Procedures and control measures to prevent accidental reaction involving the CAC waste include:

1. A dedicated Alachlor process incinerator -- no ex-process plant wastes may be burned at the facility.
2. Process/pipeline separation until the waste is fired into the burner plenum.
3. Flame out at the incinerator activates automatic shutoff of the waste feed.
4. Departmental hazardous chemical information on the waste lists the material as very reactive with water. This information is reinforced in departmental training.
5. Daily visual inspections of storage tank, piping, and incinerator [264.347(b)] and inspection data kept in the operating log [264.347(d)].

IV. DETAILED ENGINEERING DESCRIPTION OF INCINERATOR

IV.1 Manufacturer's Name and Model Number of Incinerator

The principal components of the Monsanto Chemical Company's Queeny plant CAC incinerator were designed and fabricated by the John Zink Company of Tulsa, Oklahoma. Built circa 1976 under Service Order #081181, it is a design with no model number.

IV.2 Type of Incinerator

The incinerator is designed as a combination liquid injection and gas thermal oxidizer, consisting of a horizontal burner plenum, vertical oxidizer chamber, water quench pot, and water absorber (scrubber). Waste gases are no longer burned in the unit.

Auxiliary fuel (natural gas) is used to bring the oxidizer up to minimum operating temperature (1650^{o}F) before the waste streams are introduced and maintain the correct operating temperature (1800^{o}F) under normal operating conditions. The liquid waste enters the system by way of special patented burners under pressure with auxiliary steam to assure complete atomization. When fired, a high temperature oxidizing region is formed through which the waste must pass. The waste is thermally dissociated and then oxidized with an excess of combustion air (25%). The furnace is sized to insure sufficient residence time for all reactions to go to completion. A minimum of auxiliary fuel is used to maintain stable burning temperatures for the waste streams.

The oxidizer unit is a vertically oriented, self-supported unit. Gaseous reaction products and inerts exit near the top of the oxidizer and are directed downward through a specially designed aqueous quench system. The gas stream is quenched from 1800^{o}F to 190^{o}F, then directed to a combination absorption column and vent stack. Hydrogen chloride is removed from the combustion gas stream by means of a counter-current aqueous stream in a packed absorption column.

A schematic diagram of the CAC incinerator is shown in Figure IV-1. The incinerator is a "forced draft" type unit, in that the prime mover is the combustion air blower. The general arrangement plan view of the incinerator system is shown in engineering drawing no. B-O-81181-202; the side elevation is shown in drawing no. -203; and the end elevation is shown in drawing no. -204, contained in Appendix A. The nozzle legend is given in drawing no. -201. Materials of construction for each section or element in the incineration system are given on the engineering drawings.

Key incinerator design information is summarized in Table IV-1.

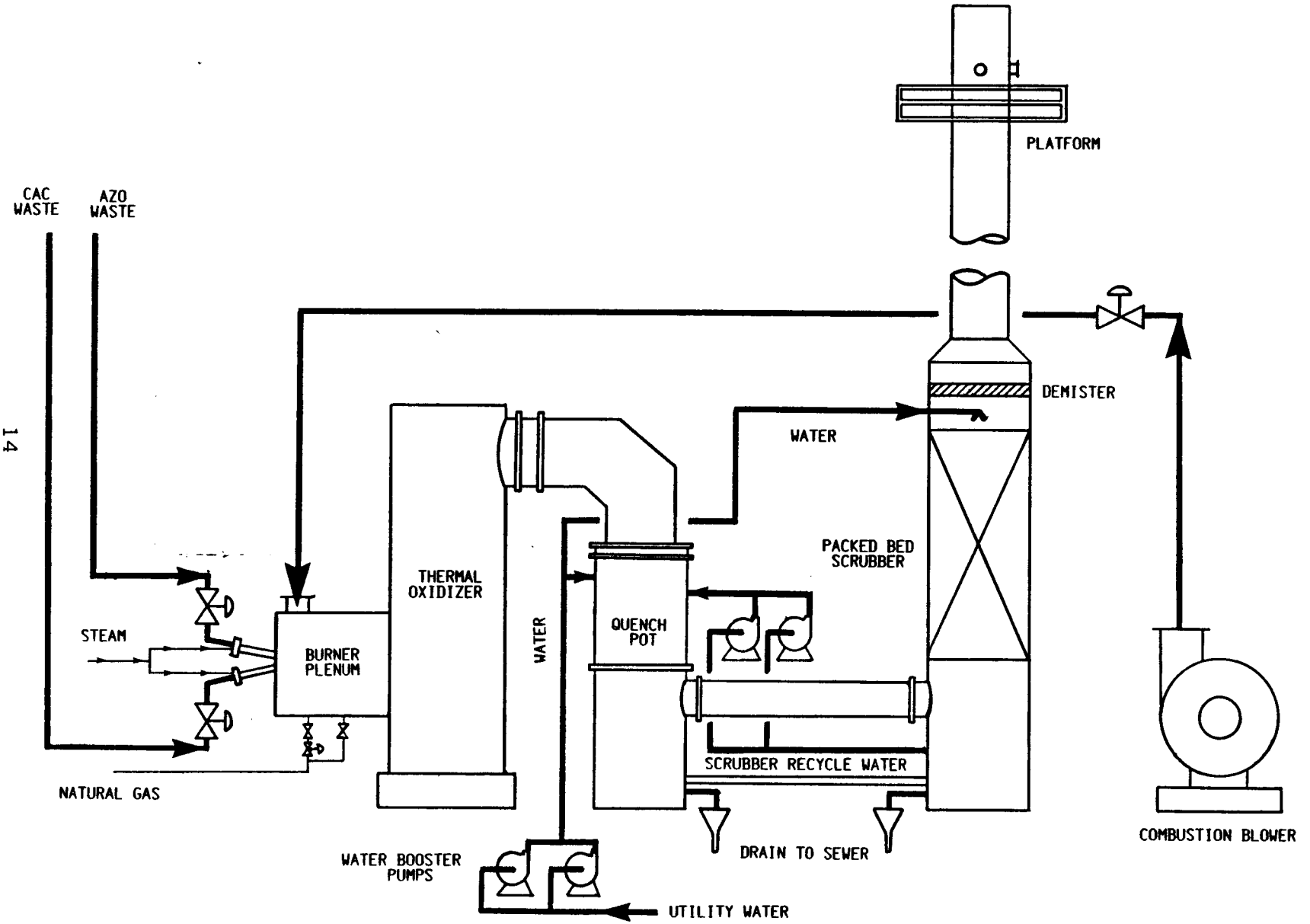


Figure IV-1. Schematic diagram of the Monsanto-Queeny plant CAC incinerator system.

TABLE IV-1. SUMMARY DESIGN INFORMATION

Parameter	Units	Combined system
Type of incinerator	--	Liquid fired burners to vertical thermal oxidizer
Inside dimensions (diameter x length)	ft	Burner plenum = 3.0 x 3.0 Oxidizer = 5.792 x 21.906 Breeching duct = 2.875 x 14.617
Cross-sectional area	ft^2	Burner plenum = 7.07 Oxidizer = 26.35 Breeching duct = 6.49
Volume, inner	ft^3	693.3
Heat capacity	10^6 Btu/hr	17.0
Refractory thickness	inches	Burner plenum = 9.0 Oxidizer = 4.5 Breeching duct = 3.0
Refractory conductivity	Btu-in./ $hr-ft^2-{}^oF$	unknown (assume 5% heat loss)
Refractory surface area	ft^2	593.8 (all surfaces)
Cooled surface area	ft^2	0
Waste feed system	--	Liquid injection with steam atomization
Installation date	year	1976
Blower/fan capacity	scfm	4000
Maximum quench inlet temperature	^oF	2000
Maximum scrubber inlet temperature	^oF	190
HCl removal capacity	lb/hr	540 (1400 lb/hr @ 39% Cl)
Quench water supply capacity	gpm	Utility water = 100 Scrubber recycle = 75
Quench water temperature	^oF	Utility water = 60 Scrubber recycle = 176

V.3 Linear Dimensions of the Incinerator Unit Including the Cross-Sectional Area of the Combustion Chamber

The incinerator section of the system consists of a burner plenum followed by a thermal oxidizer chamber. The burner plenum, shown in drawing no. -305, has outside dimensions of 4.33 ft. in diameter by 6.0 ft. in vertical length. Inside the plenum, combustion air enters through a 1.208 inch diameter tangential duct, where it meets 20 spin vanes. Waste burner guns enter vertically at angles, shown in drawing no. -601, through the end of the plenum and extend to the refractory tile section of the plenum. The plenum refractory section consists of an 11 inch I.D. entry to a cylindrical chamber which is 3.0 ft. long by 3.0 ft. I.D., and exit through a 9 inch long by 2.042 inch I.D. connecting duct. The refractory tiles are C.E. Chemal 85B fire brick.

The natural gas pilot assembly protrudes into the front of the tiled plenum along with scanner/control nozzles and an auxiliary gas nozzle. The gas pilot assembly is shown in drawing no. -608.

The thermal oxidizer section stands vertically and is 23.344 ft. high by 7.0 ft. in diameter outside dimensions. The inlet duct is centered 3.344 ft. above the chamber outside floor. Inside dimensions are 22.014 ft. high by 5.792 ft. in diameter for the refractory lined oxidizer chamber, as shown in drawing no. -306. Gases leave the oxidizer through a 2.875 ft. I.D. refractory lined breeching duct near the top of the chamber. Other connections and nozzles to the thermal oxidizer chamber include water/steam tempering guns (see drawing no. -602), sight port, waste gas feed nozzle (not used), and manway. Refractory limits are rated at 2900 ^{o}F.

IV.4 Description of the Feed and Fuel Burner Systems

The waste feed to the incinerator goes through the John Zink Model HI-24 burner assembly, shown in drawing no. -601. Both CAC Azo residues are fed to the burner plenum using a "DH" waste gun insert, shown in drawing no. -607. CAC residue is fed at 1100 lb/hr (about 1.5-1.6 gpm) liquid at 65 psig at the gun tip, with atomizing steam at 90 psig. The CAC waste recirculation line carries about 60 gpm back to the CAC residue storage tank. When fired, Azo residue is fed at 275 lb/hr liquid at 50 psig at the gun tip, with atomizing steam at 80 psig. Provision is made in the HI-24 assembly for two other burner guns, one for auxiliary fuel oil (not used) and another for a former liquid waste stream (not used). Both burner guns are the John Zink "EA" oil type gun, shown in drawing no. -605. The "EA" oil guns are rated for #2 fuel oil, 100 psig available pressure, 70 psig oil pressure at the tip, with atomizing steam at 100 psig.

The auxiliary fuel gas enters the burner plenum through the side, shown in drawing no. -305, adjacent to the gas pilot

assembly. The pilot is rated at 380,000 Btu/hr natural gas, and the auxiliary gas burner is rated at 5,500,000 Btu/hr. Fuel gas specifications include 910 Btu/scf, 0.55 specific gravity, 30 psig available, and 10 psig at the burner.

IV.5 Capacity of Prime Mover

The combustion air blower is a Garden City Blower Model No. 325-6-40 powered by a 40 hp electric motor operated at 250 rpm to push the combustion gases through the incinerator system. Combustion air requirements are 3300 scfm at 30 in. H_2O static pressure and 100°F ambient with 25% excess air combustion requirement. This blower has a nominal capacity of 4000 scfm at 100°F. The combustion air flow can be c ontrolled by a gate valve, which is instrumented to respond automatically or manually to a low flow switch.

IV.6 Description of the Quench System

From near the top of the oxidizer, the exhaust gases are directed out and down to the quench pot through a 3.583 ft. O.D. refractory-lined breeching duct (2.875 ft. I.D.), shown in drawing no. -304. The quench pot, shown in drawing no. -301, supports a contact tube, water weir and aqueous quench gun assembly, shown in drawings nos. -302 and -606. Approximately 50 gpm of water are used to quench the exhaust gases through eight quench water guns located on the quench contactor circumference, four each at two levels. The upper ring of quench guns uses water supplied from the boosted utility water header, and the lower quench ring uses water supplied from the scrubber recycle pumps, as shown in Figure 1. The quench contactor section is 4.0 ft. high by 4.0 ft. I.D., and is refractory-lined to a final inner diameter of 3.25 ft.

The quench pot is 4.0 ft. I.D. by 9.604 ft. high, with a downcomer that extends 2.937 ft. into the pot section. Cooled exhaust gases leave the quench pot near the top past the downcomer through a 2.167 ft. I.D. duct. Quench water is discharged from the quench pot through a drain connection (nozzle #23) to the plant sewer.

IV.7 Description of the Scrubber/Absorber

A 2.167 ft. I.D. by 16.833 ft. connecting duct, shown in drawing no. -309, routes exhaust gases from the quench pot to the packed tower absorber. The lower absorber section, shown in drawing no. -307, is 5.0 ft. I.D. by 26.5 ft. high. From the 10.5 ft. high mark to the 20.0 ft. mark (9.5 ft.) is the packed tower absorber section, consisting of a Norton #22808 ceramic packing support plate, 59 inches O.D., and 200 ft^3 of 2-inch tel-zell Glitsch saddles (1/8-inch wall thickness). 140 gpm of water flows through a 3-inch teflon-lined exterior nozzle pipe directed down onto a Norton titanium distributor above the packed tower section. Just above the scrubber water nozzle is a York Durakane Model #470-45 mist eliminator pad, shown in drawing no.

A-ST-0001. Scrubber water is discharged from the lower absorber section through a drain connection (nozzle #27) to the plant sewer. The absorber and quench pot are connected by a 4-inch line for liquid level equalization.

The absorber upper section acts as the incinerator stack to vent the cleaned exhaust gases to atmosphere, shown in drawing no. -308, and is necked down from 5.0 ft. I.D. to 2.167 ft. I.D. at the 28 ft. high mark. Stack exit is 50.0 ft. above ground level. Sampling and gas monitoring ports are located at 45.0 ft. above ground level with a service platform, shown in drawing no. -310, surrounding 300° of the circumference and located at 40.0 ft. above ground level.

IV.8 Location and Description of Temperature, Pressure, Level and Flow Indicating and Control Devices

The process control sensors and monitors described in Table IV-2 are used at the facility. The location of the sensors is shown in Figure IV-2; more detailed location information can be found by reviewing the appropriate engineering drawing for a system component or section in Appendix A.

Note: Engineering projects are underway for design, purchase, and installation of stack gas CEMS and upgrade of the waste feed automatic shutoff system.

TABLE IV-2. SUMMARY OF CAC INCINERATOR PROCESS MONITORS

Parameter	Location of monitor[a]	Type of monitor	Operating range	Units recorded
CAC residue feed rate	1	Mass flowmeter	0-3000	lb/hr
Azo residue feed rate	2	Mass flowmeter	0-600	lb/hr
Atomizing steam	3	Pressure gauge	0-110	psig
Auxiliary fuel gas flow rate	4	Turbine meter	0-320	lb/hr
Combustion air flow rate	5	Valve setting calculation	0-4000	acfm
Burner flame	6	UV detector	--	flame failure
Thermal oxidizer temperature (4)	7	Thermocouple (Type K)	500-1500	°C

TABLE IV-2. SUMMARY OF CAC INCINERATOR PROCESS MONITORS (Continued)

Parameter	Location of monitor[a]	Type of monitor	Operating range	Units recorded
Quench H_2O flow rate	8	Magnetic flowmeter	0-100	gpm
Quench pot temperature (3)	9	Thermocouple (Type J)	0-200	^oC
Scrubber recycle flow rate	16	Magnetic flowmeter	0-100	gpm
Scrubber/absorber inlet gas temp	10	Thermocouple (Type J)	0-200	^oC
Scrubber water flow rate	11	Magnetic flowmeter	0-200	gpm
Scrubber/absorber water level	12	DP cell	0-100	%
Utility water boosted pressure	13	Pressure gauge	0-200	psig
Oxygen (to be installed)	14	Thermomagnetic	0-21	%
Carbon Monoxide (to be installed)	15	extractive NDIR	0-100 0-2000	ppm ppm

[a] Refer to Figure IV-2.

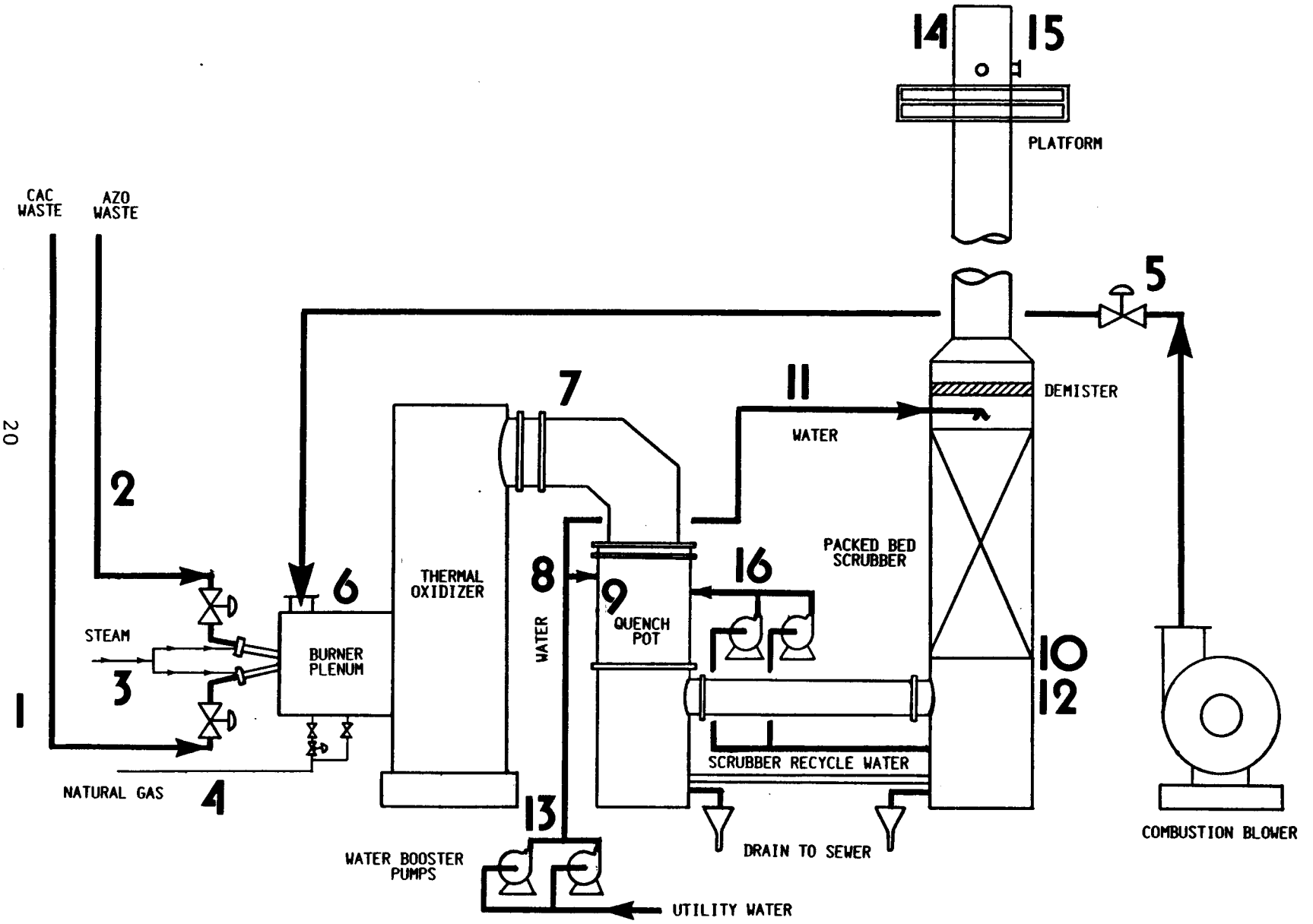


Figure IV-2. Location of CAC incinerator process control sensors and monitors (see Table IV-2).

IV.9 Description of Automatic Waste Feed Cut-off System

Instrumentation is provided to monitor incineration process conditions, provide data for assuring compliance with regulations, and assure appropriate process response and control, operational flexibility, and safety interlocking and shutdown features. The safety interlocks and shutdown features comprise a major portion of the control system.

A summary of the process monitors was given in Table IV-2. Safety shutdown responses are relayed to various equipment items when process limits are not met; these include additional safety features beyond simple waste feed cut-off. In general, the process parameters that alert and initiate responses to alarm conditions are described in Table IV-3. The interlock system is configured to first alarm and then shutdown components of the incinerator system.

Figure IV-3 is a schematic diagram of the instrumentation surrounding the oxidizer section of the CAC incinerator system. A description of each of the interlock systems is given below:

A. INTERLOCK #10

1. This interlock is tripped by:
   a. High oxidizer temperature, TSH 114-51, TSH 114-52, TSH 114-7.
   b. Low natural gas pressure, PSL 114-16, PSL 114-29, activated on a time delay.
   c. Low water flow to quench and/or scrubber, FSL 178-3, FSL 178-4, FSL 178-5.
   d. Manual trip.

2. This interlock causes the following action:
   a. Stops flow of all liquid wastes to incinerator.
   b. Stops flow of natural gas to incinerator, BV 114-11, BV 114-13, SV 114-24, SV 114-25, SV 114-26.

C. INTERLOCK #23

1. This interlock is tripped by:
   a. Low oxidizer temperature (manual reset with lock), TSL 114-1.

2. This interlock causes the following action:
   a. Stops flow of liquid wastes to incinerator.

D. INTERLOCK #25

1. This interlock is tripped by:
   a. Low steam pressure on waste guns, PSL 114-36.

2. This interlock causes the following action:
   a. Stops flow of waste to incinerator, FICA 156-5.

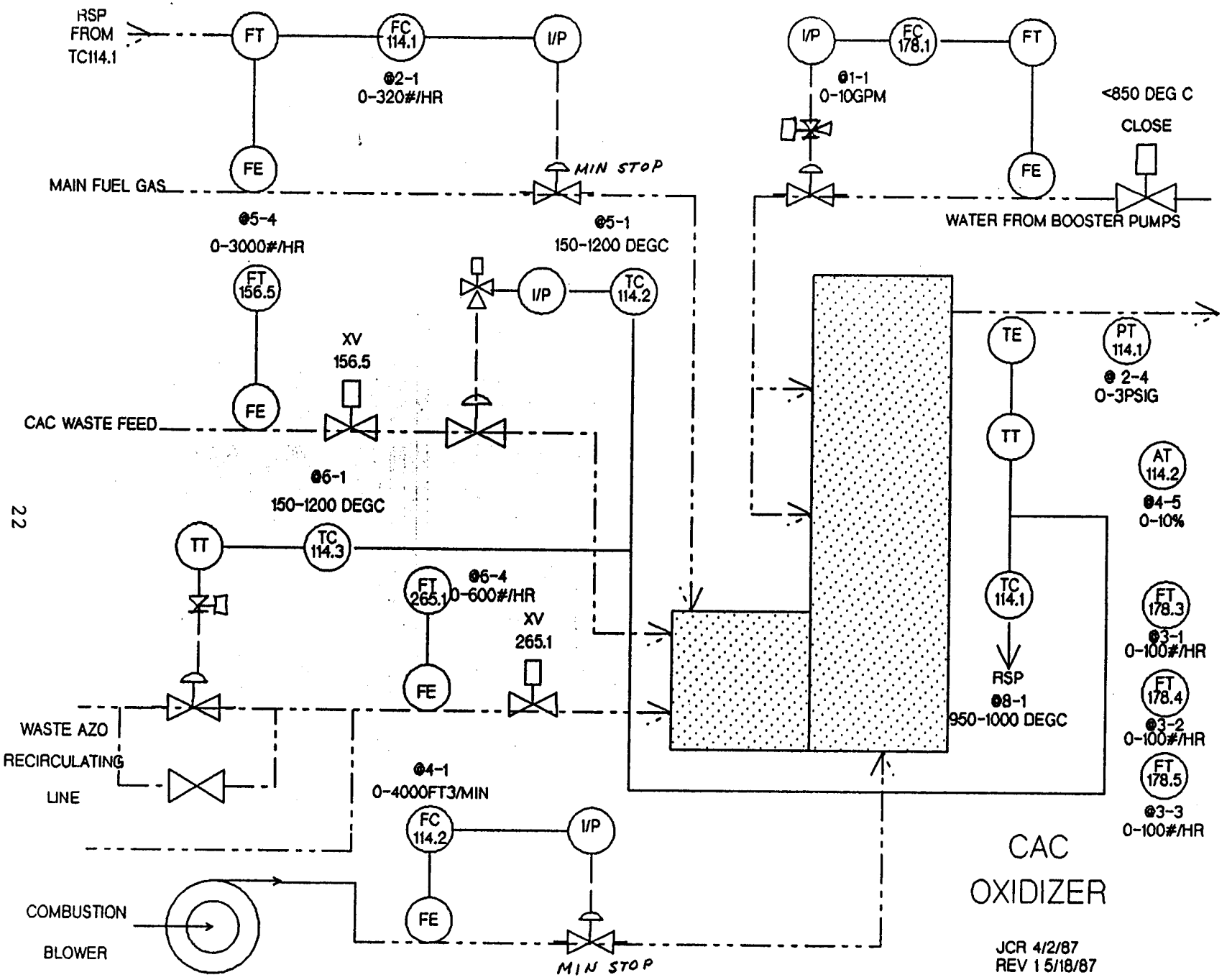


Figure IV-3. Schematic diagram of instrumentation for the CAC oxidizer section.

E. POWER FAILURE

A 480 volt power failure causes the same action to occur as does Interlock #10 when it is tripped.

Similarly, a thermocouple failure for one of the high (fails upscale) or low temperature set points causes the same action to occur as does Interlock #10 when tripped.

F. FLAME SAFEGUARD SYSTEM

1. This system is triggered by:
   a. Loss of pilot flame, BS 114-3.1.
   b. Loss of main flame, BS 114-3.2.

2. When triggered, this system causes the same action to occur as does Interlock #10 when it is tripped.

G. START-UP OVERRIDES

There are overrides designed for startup procedures, to allow the incinerator to be brought up to temperature on natural gas only.

Interlock overrides for start-up are provided for the following switches: BS 114-3.1, BS 114-3.2, TSL 114-1, PSL 114-29, PSL 45.

Since either natural gas or fuel oil can be used for auxiliary fuel, provisions are made to override PSL 114-16 or PSL 114-45 when that particular fuel is not being used. Note: gas only is used.

Other procedures for the incinerator such as 1) initial refractory curing instructions, 2) normal cold start-up after initial refractory cure-out, 3) normal shutdown, 4) emergency shutdown, 5) start-up procedure, 6) normal start-up checklist, and 7) troubleshooting guideline are contained in Appendix C - Incinerator Procedures.

Alarm testing is conducted on a weekly basis in accordance with 40 CFR 264.347(c).

TABLE IV-3. SUMMARY OF CAC INCINERATOR INSTRUMENT SET POINTS AND FUNCTION

Item	Number	Normal	Interlock set point	Alarm set point
Low gas pressure-Main	PSL 114-29	10 psig	3 psig	4 psig
High gas pressure-Main	PSH 114-28	10 psig	16 psig	15 psig
Low gas pressure-Pilot	PSL 114-16	10 psig	3 psig	4 psig
High gas pressure-Pilot	PSH 114-15	10 psig	16 psig	15 psig
Low atomizing steam pressure	PSL 114-32	100 psig	--	45 psig
CAC waste-low atomizing steam pressure	PSL 114-36	90 psig	20 psig	45 psig
Azo waste-low atomizing steam pressure	PSL 114-38	80 psig	20 psig	45 psig
Low water weir flow	FSL 178-4	25 gpm	5 gpm	10 gpm
Low quench recycle water flow	FSL 178-5	50 gpm	5 gpm	10 gpm
Low quench fresh water flow	FSL 178-4	50 gpm	5 gpm	10 gpm
Low scrubber water flow	FSL 178-3	140 gpm	30 gpm	40 gpm
Incinerator scrubber pump	ESL 135-1	On	--	Down
Incinerator water booster pump	ESL 178-6	On	--	Down
Low combustion air flow	FSL 169-2	3000 scfm	--	750 scfm
Low CAC waste flow	FICA 156-5	1100 lb/hr	--	400 lb/hr
Low oxidizer temperature	TIC 114-1	980°C	870°C	900°C
High oxidizer temperature	TSH 114-7	980°C	1090°C	1070°C

TABLE IV-3. SUMMARY OF CAC INCINERATOR INSTRUMENT SET POINTS AND FUNCTION (Continued)

Item	Number	Normal	Interlock set point	Alarm set point
High quench pot temperature	TSH 114-51 TSH 114-52	88°C	140°C	135°C
Pilot gas flame failure	BA 114-3.1	On	Flame failure	Flame failure
Main gas flame failure	BA 114-3.2	On	Flame failure	Flame failure
Low absorber/quench water level	LR 114-5	50 %	--	25 %
High absorber/quench water level	LR 114-5	50 %	--	75 %
Low stack gas O_2 (to be installed)				To be set in permit
High stack gas CO (to be installed)				To be set in permit

IV.10 Stack Gas Monitoring Equipment

The stack gas monitoring equipment will be extractive analyzers. The extraction system will consist of a Hastelloy sample probe coupled to a Westinghouse sample conditioner for gas sample cleanup. Heat tracing of the sample line from the probe to the analysis system is provided for freeze protection as the sample conditioner is designed for high condensate formation. The interconnecting lines will be constructed of teflon.

The wet particulate-laden sample is processed initially through a coarse bypass filter assembly, consisting of a plastic water knockout pot and drained by an eductor with an integral water condensate manual flush valve. 90% of the condensate is removed in this stage. Blowback of the sample line is accomplished by a large orifice PVC 3-way air operated ball valve directly adjacent to the coarse bypass filter assembly. This valve is programmed automatically to periodically blowback the

sample through the open probe.

Closely coupled to the coarse bypass filter assembly is a Hastelloy heat exchanger which reduces the sample dewpoint rapidly to approximately 40°F. Once the gas sample has been chilled, it passes in to an additional educted fine bypass filter assembly and the dry sample passes through a coalescing PVC filter element to prevent any acid condensate or water droplets from being carried to the balance of the system. The sample is drawn from the fine bypass filter assembly, through a surge reservoir, and to the two Westinghouse analyzers. This method of sample transport insures that the analyzers receive a sample under positive pressure and, therefore, can be vented to atmosphere to eliminate calibration problems.

The CO analyzer is a Westinghouse Model No. UNOR 6N NDIR analyzer with dual ranges from 0-100 ppm and 0-1000 ppm. Its specifications include zero drift of <1% of full scale per week, span drift of <1% of full scale per week, repeatability of <0.2% of full scale, and linearity of <1% of full scale.

The O_2 analyzer is a Westinghouse Model No. Oxygor 6N thermomagnetic analyzer with a range of 0-21%. Its specifications include zero drift of <1% of full scale per week, span drift of <2% of full scale per week, repeatability of <0.5% of full scale, and an MDL of 0.2% of full scale.

IV.11 Security Procedures for the Incinerator Facility

The incinerator facility includes plant perimeter fencing with gates and guards, as discussed elsewhere in the Part B permit application.

Upon entering the CAC Intermediate process area in which the incinerator is located, visitors (nonprocess employees) are required to first stop in the Control Room to inform the process operator and/or foreman that he will be out in the process area. The incinerator area boundaries are marked with signs which require wearing of goggles inside the incinerator process area, in addition to the standard plant requirements of hard hats, safety glasses, and steel-toed shoes.

Warning signs, which are legible from a distance of 25 ft., are posted at several locations around the incinerator. These signs are visible from all angles of approach and bear the legend "DANGER - UNAUTHORIZED PERSONNEL KEEP OUT".

IV.12 Inspection Requirements

The CAC incinerator system personnel conduct routine inspections of the incinerator and auxiliary equipment for malfunctions, structural deterioration, operator errors, leaks, spills, and signs of tampering. The general inspection schedule and inspection log sheets have been provided in that section of the Part B permit application.

V. TRIAL BURN PROTOCOL

The objective of the trial burn is to demonstrate compliance of the CAC incinerator system with the incinerator standards sa promulgated in 40 CFR 264, Subpart O. The trial burn is structured to first identify the operating conditions for the incinerator necessary to assure compliance with the applicable standards, and secondly to establish an operating range in which the standards are achieved. The trial burn proptocol is also designed to present sufficient data regarding the incinerator system performance to expedite the permitting process.

Under a series of operating conditions during testing, the trial burn will demonstrate attainment of the three primary performance criteria:

1. Stack gas particulate emission rate not to exceed 180 mg/dscm (0.08 gr/dscf), when corrected to 7% oxygen (O_2).

2. Attain a hydrogen chloride (HCl) emission rate not to exceed 1.8 kg/hr (4 lb/hr) or greater than 99% HCl removal efficiency.

3. The destruction and removal efficiency (DRE) for selected Principal Organic Hazardous Constituents (POHCs) in the waste feed must be at least 99.99%.

The test protocol has been developed using proven and accepted test methods and techniques for the appropriate parameters (see Section VI). A quality assurance/quality control program plan has also been incorporated into the testing protocol to ensure known precision, accuracy, and completeness of the data (see Section VII).

V.1 POHC Selection

Chemical composition analyses of the CAC waste was presented in Section III. The CAC waste is the only hazardous waste stream burned in the CAC incinerator, although Azo residue is burned when CAC waste is not. Eight (8) compounds in the CAC waste have been identified as 40 CFR 261, Appendix VIII hazardous constituents, and can qualify as POHCs in the trial burn. These constituents and their respective weight percents in CAC waste are: 1) acetyl chloride, 34.5% (CH_3COCl); 2) ethylene dichloride or 1,2-dichloroethane, 1.8% (CH_2ClCH_2Cl); 3) 2,3-dichloro-1-propene, 0.044% ($CH_2=CClCH_2Cl$); 4) chloroform, 0.013% ($CHCl_3$); 5) chloromethane, 0.0088% (CH_3Cl); 6) carbon tetrachloride, 0.0067% (CCl_4); 7) tetrachloroethane, 0.0037% ($CH_2Cl_2CH_2Cl_2$); and 8) dichloromethane or methylene dichloride, 0.0035% (CH_2Cl_2).

There are three methods used to prepare the desired quantity of waste possessing the desired characteristics for the trial burn protocol. The three methods are:

1. Use actual wastes;

2. Use a prespiked or synthetically prepared wastes; or

3. Continuously spike POHC(s) into the waste during the test condition.

We propose to spike a compound of difficult "incinerability" into the CAC waste -- tetrachloroethylene (CAS# 127-18-4) -- to offset the infeasibility of sampling and analyzing for trace concentrations of acetyl chloride in a saturated exhaust gas stream. The tetrachloroethylene will be spiked into the CAC residue storage tank at a concentration level of about 2.0%, and kept well mixed via continuous feed pump recirculation before the testing. Samples of CAC waste will collected during each test condition and analyzed to measure the true spike concentration for DRE calculation purposes.

Table V-1 presents the POHCs considered for testing and their relative ranking on two "incinerability" indices -- the heat of combustion index and the University of Dayton Research Institute (UDRI) thermal stability index. The UDRI index is based on measured and <u>estimated</u> gas-phase thermal stabilities for a 99% destruction at 2.0 seconds gas-phase residence time [$T_{99}(2)(^{o}C)$]. Please note that <u>none</u> of the POHCs listed in Table V-1 have been evaluated on the UDRI thermal decomposition system.

There are two compelling reasons for eliminating acetyl chloride from the POHC selection for trial burn testing -- the compound is highly water reactive, thus readily destroyed/removed from the incineration system, and there exists no validated measurement methodology for trace quantities of the compound in stack gas.

For an acid or base, the equilibrium or ionization constant is an indicator of the strength of an acid, or its ability to dissociate in aqueous media. Very strong dissociators, such as sulfuric acid (H_2SO_4) or hydrochloric acid (HCl) have large ionization constants in the range of 10^{+3} or 10^{+5}, respectively. Another measure is the rate of hydrolysis, the chemical transformation process in which an organic molecule reacts with water, forming a new carbon-oxygen bond (R-OH) and cleaving a carbon-X (chloride) bond in the original molecule. Hydrolysis of organic chemicals in water is first-order in the concentration of organic species; the rate of disappearance of RX, -d[RX]/dt, is directly proportional to the concentration of the compound, [RX]:

$$- d[RX]/dt = k_T[RX]$$

where k_T = hydrolysis rate constant. The hydrolysis half-life is then:

$$t_{1/2} = 0.693/k_T$$

The Handbook of Chemistry and Physics, 54th edition (1973), CRC Press, Cleveland, Ohio, does not list an ionization constant for acetyl chloride; instead, it simply states that acetyl chloride decomposes in water. The hydrolysis constant for acetyl chloride is estimated to be 1.1, based on procedures in the Handbook of Chemical Property Estimation Methods, edited by W. J. Lyman (1982). The estimated half-life in water is then 0.7 seconds.

No measurement method exists in EPA-SW-846 for determining acetyl chloride concentration in a waste sample, much less chance for determining trace quantities in a saturated gas stream. Review of methods which may be applicable shows that EPA 8010 (gas chromatography for halogenated volatile organics), EPA 8120 (gas chromatography for chlorinated hydrocarbons), and EPA 8240 (GC/MS for volatile organics) do not list acetyl chloride as an analyte for which the method is applicable. In sampling stack gas, any collection media (sorbent, charcoal, liquid absorbent) will be exposed to a water-saturated gas stream which would react away any trapped acetyl chloride. Any derivatization techniques suffer from nonspecificity to the acetyl or chloride ions present in other constituents in the CAC waste. An in-situ or real-time monitoring method for acetyl chloride, if one existed, would suffer from the need to remove moisture from the stack gas sample, thus degrading the sample.

Tetrachloroethylene was chosen as a POHC to be spiked into the CAC waste because of the following reasons:

1. It has a "incinerability" ranking at the top of the list based on its heat of combustion index, as shown in Table V-1.
2. It is compatible with the CAC waste and should be readily miscible.
3. Although carbon tetrachloride is present in ppm levels in the CAC waste and could be spiked up to percent levels, tetrachloroethylene was chosen because it has ten times the breakthrough volume on the sorbent Tenax GC, and will allow for longer safe sampling periods.

TABLE V-1. POHC SELECTION CRITERIA SUMMARY

Hazardous Constituent	Ranking		
	Heat of Combustion (kcal/g)	UDRI Thermal Stability T_{99}(2)	Choice?
Tetrachloroethylene spiked to 2.0% (CAS# 127-18-4)	15th (1.19)	26-28, Class 2	Yes
Ethylene dichloride actual about 1.8% (CAS# 107-0602)	62nd (3.00)	170-173, Class 6	Yes
Acetyl chloride actual about 34% (CAS# 75-36-5)	51st (2.77)	65-68, Class 3	No
Dichloropropene, N.O.S. actual about 0.04% (CAS# 26952-23-8)	76th	84-85, Class 3	No

V.2 Test Conditions, Maximums, and Minimums

Three test conditions are proposed during the trial burn to demonstrate the performance of the incinerator system under a useful range of operating conditions. The test conditions represent the typical range of operation for the CAC incinerator, including two "extreme" conditions used to establish maximum and minimum parameters for setting permit limits.

During each of the three test conditions, triplicate test runs for sampling/analysis and process parameter data collection will be performed, providing a total of nine sets of data.

During the first test condition, CAC waste will be fired in the incinerator at "expected normal" operating rates. The total waste feed rate will be targeted at 1100 lb/hr.

During the second test condition, those conditions reflecting "low" rates for burning CAC waste will be tested with CAC waste at about 800 lb/hr; in reality, the target oxidizer operating temperature of 900-950^{o}C is set according to the CAC waste feed rate. This test condition will establish minimum oxidizer temperature, minimum total heat input, and minimum quench and scrubber water flow rates.

During the third test condition, CAC waste will be burned at an approximate rate of 1400 lb/hr; again, set according to the target oxidizer operating temperature of 1050-1080^{o}C. This test condition represents a "maximum", and will establish the highest concentration of POHCs, the maximum waste feed rates, maximum combustion air flow rate (minimum residence time), maximum CO level in stack gas, minimum O_2 level in stack gas, maximum heat input, maximum Cl feed rate, and maximum ash content feed rate.

Table V-2 presents the summary of parameters to be varied during the proposed test conditions and the indentification of which test condition contains parameter maximums or minimums for later establishment of permit conditions.

V.3 Schedule

It is anticipated that the trial burn will require nearly two weeks of on-site work by the test crew. The on-site work will include:

1. Preparation of a laboratory and sample recovery area;
2. Set-up of sampling equipment at the test site;
3. Briefings for plant safety, emergencies, test protocol, personal protective equipment, and company-contractor coordination;
4. Preliminary measurements of the stack gases for cyclonic flow check, temperature, velocity, and

moisture;
5. The test period; and
6. Site clean-up, equipment packing, preparation of samples for travel, and completion of QA/QC details.

Two days will be allocated for each test condition, in which the required triplicated test runs will be completed. The actual test period will be preceded by a time in which the incinerator conditions are allowed to stabilize to the target levels. Testing will be followed each day by sample recovery, labeling, secure storage, checks for QC and completeness, and preparation of the sampling equipment for the next test run.

The estimated schedule for conducting the trial burn, analyzing the collected samples, preparing the final report, and submitting results to the regulatory agencies is shown in Table V-3.

V.4 **Waste Quantities**

Each test condition has been assumed to require a maximum twenty (20) hours of waste burning to to allow ample time for completing the triplicate test runs (3-4 hours). Table V-4 presents the summary of waste quantities proposed to be burned during the trial burn performance tests.

TABLE V-2. SUMMARY OF TEST CONDITION PARAMETER TARGETS FOR CAC INCINERATOR SYSTEM

Incinerator system parameter	Test condition 1	2	3
Waste type(s)	CAC	CAC	CAC
Operation mode	normal	low	maximum
Waste feed rate (lb/hr)	1100	800[a]	1400[a]
POHC feed rate (lb/min) Perc	0.367	0.267	0.467
EDC	0.330	0.240	0.420
Chlorine feed rate (lb/hr)	407	296	518
Ash feed rate (lb/hr)	TBD	TBD	TBD
Waste heat input (mmBtu/hr)	9.086	6.608	11.564
Auxiliary fuel (mmBtu/hr)	2.275	2.275	2.275
Combustion air (scfm)	3300	3300	3300
Oxidizer temperature (^{o}C)	900-950	980-1040	1050-1080
O_2 in stack gas (%)	TBD[b]	TBD	TBD
CO in stack gas (ppmv)	TBD	TBD	TBD
Quench water flow (gpm)	25	25	25
Scrubber water flow (gpm)	140	140	140
Scrubber recycle flow (gpm)	50	50	50

[a] Will become set according to oxidizer temperature.
[b] To be determined during trial burn tests.

TABLE V-3. SCHEDULE FOR CAC INCINERATOR TRIAL BURN COMPLETION

Event	Day number
1. Final approval of trial burn plan by Missouri DNR and EPA Region VII	1
2. Initiate trial burn testing	within 90
3. Conduct trial burn testing	90-100
4. Analyze samples; prepare final report	100-160
5. Submit final report to agaencies	161

TABLE V-4. SUMMARY OF WASTE QUANTITIES TO BE BURNED DURING MONSANTO-QUEENY CAC INCINERATOR TRIAL BURN PERFORMANCE TESTING

	Test condition			
Waste type	1	2	3	Total
CAC residue, lbs.	16,000	22,000	28,000	66,000

VI. SAMPLING AND ANALYSIS PLAN

The sampling and analysis plan is designed to quantitatively measure the input and output of selective environmental parameters with the purpose of demonstrating the attainment of regulatory incinerator performance standards. Input streams to be measured include the waste feeds, utility water, and combustion air; output streams to be measured include quench water discharge, scrubber water discharge, stack gas flow and composition, and fugitive emission screening. Figure VI-1 shows the sampling locations to be used at the CAC incinerator.

The subcontractor chosen to conduct the sampling and analysis program will be PEI Associates, Inc. of Cincinnati, Ohio.

An overview of the sampling program is summarized in Table VI-1, with standardized EPA methods designated by identification numbers given either in 40 CFR 60 Appendix A, EPA-SW-846, or EPA-600/8-84-002 (ampling and Analysis Methods for Hazardous Waste Combustion).

An overview of the analytical program is summarized in Table VI-2, with standardized EPA methods designated by identification numbers given either in 40 CFR 60 Appendix A, EPA-SW-846, EPA-600/4-84-017 (Methods for Chemical Analysis of Water and Wastes), or American Society for Testing and Materials (ASTM).

VI.1 Waste Feed

Waste feed samples of CAC residue will be collected every 30 minutes in precleaned glass bottles, and then composited for each test run. Special personal protective equipment and procedures are required for CAC waste, and have been described in the Waste Analysis Plan. Figure VI-2 illustrates the sample collection location for the CAC waste pipe line.

Analysis for POHCs in the CAC waste will be done by dilution of the waste sample in methanol (EPA 3580) to a concentration level suitable for volatiles analysis by GC/MS (EPA 8240). External analytical standards of ethylene dichloride and tetrachloroethylene will be prepared solutions in methanol by using a minimum of a zero point (blank methanol) plus four upscale calibration points. The internal standard will be d_6-benzene.

The CAC waste is anticipated to contain low to negligible concentrations of metals of concern (As, Cr, Cd and Pb), although the waste will be analyzed for these metals, nevertheless. Analysis will follow EPA-SW-846 standardized procedures.

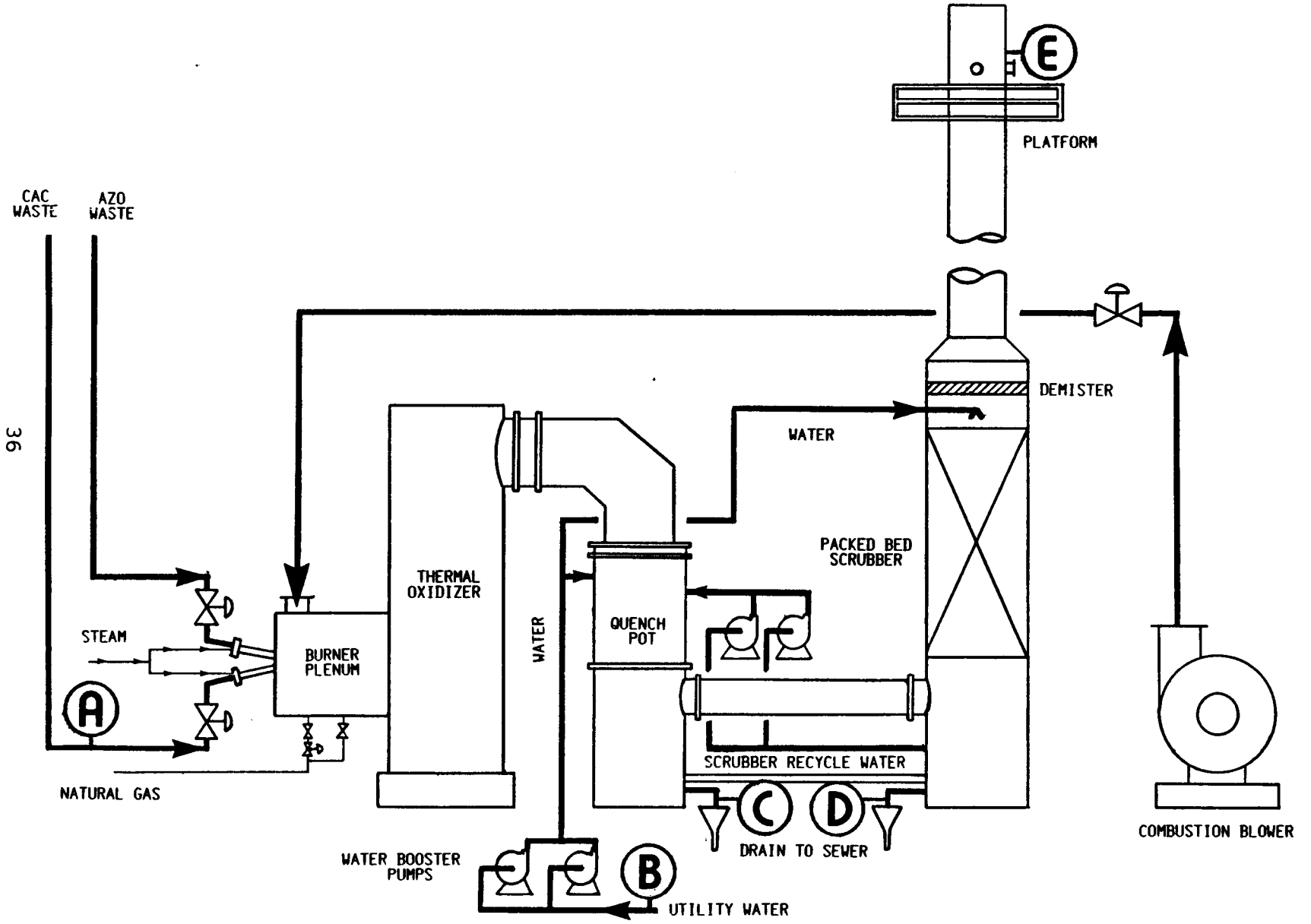


Figure VI-1. Schematic diagram of sampling locations.

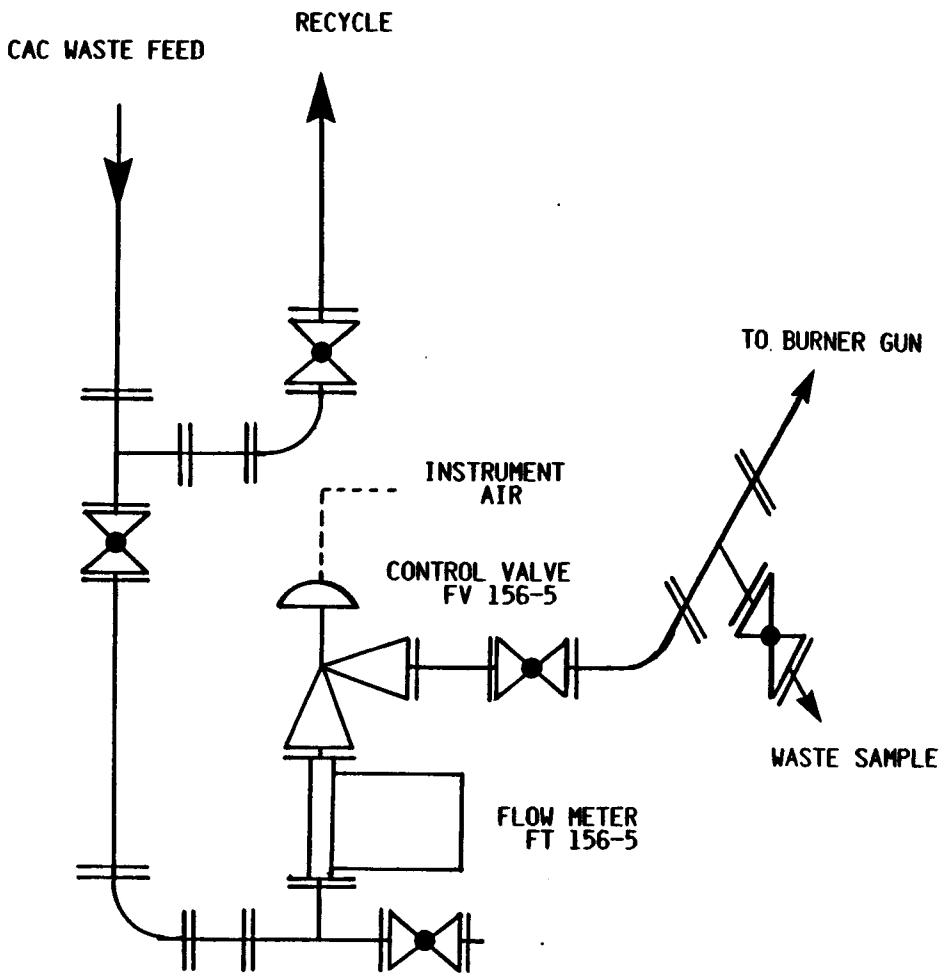


Figure VI-2. Diagram of the CAC residue sampling location.

TABLE VI-1. SUMMARY OF SAMPLING METHODS, CAC INCINERATOR TRIAL BURN, MONSANTO-QUEENY PLANT, ST. LOUIS, MISSOURI

Sample	Sample location[a]	Sampling frequency for each run	Sampling method	Sample size
CAC residue	A	One grab sample every 30 min; composited into one sample for each run	S004 (Tap)	100 mL to 1L
Utility water	B	One grab sample every 30 min; composited into one sample for each run	S004 (Tap)	40 mL per vial and 1L
Quench effluent	C	One grab sample every 30 min; composited into two samples for each run	S002 (Dipper)	40 mL per vial and 200 mL to 1L
Scrubber effluent	D	One grab sample every 30 min; composited into two samples for each run	S002 (Dipper)	40 mL per vial and 200 mL to 1L
Stack gas	E	2-hr traverse per run	EPA 5; EPA 4 and HCl in the back half	60 ft^3 minimum
Stack gas	E	2-hr traverse per run (probe attached to EPA 5 probe)	EPA 3 integrated	60 L minimum

TABLE VI-1. SUMMARY OF SAMPLING METHODS, CAC INCINERATOR TRIAL BURN, MONSANTO-QUEENY PLANT, ST. LOUIS, MISSOURI (Continued)

Sample	Sample location[a]	Sampling frequency for each run	Sampling method	Sample size
Stack gas	E	Three 40-min tests using Tenax trap pairs	EPA 0030 (VOST)	20 L minimum
Stack gas	E	Continuous	EPA 3A; EPA 10	N/A[b] N/A
Fugitive emissions	-[c]	One time survey	EPA 21	N/A

[a] Refers to Figure VI-1.
[b] Not applicable.
[c] All applicable sources.

TABLE VI-2. SUMMARY OF ANALYSIS METHODS, CAC INCINERATOR TRIAL BURN, MONSANTO-QUEENY PLANT, ST. LOUIS, MISSOURI

Sample	Analytical parameters	Preparation method	Analysis method
CAC residue	POHCs	EPA 3580 dilution	EPA 8240 GC/MS
	Metals - Pb	EPA 3050 acid digest	EPA 7060 GFAAS
	- Cd	EPA 3050 acid digest	EPA 6010 ICP
	- Cr	EPA 3050 acid digest	EPA 6010 ICP
	- As	EPA 3050 acid digest	EPA 7421 GFAAS
	Heat value	N/A	ASTM D240 Calorimetry

TABLE VI-2. SUMMARY OF ANALYSIS METHODS, CAC INCINERATOR TRIAL BURN, MONSANTO-QUEENY PLANT, ST. LOUIS, MISSOURI (Continued)

Sample	Analytical parameters	Preparation method	Analysis method
	Moisture	N/A	ASTM D95 xylene codistill
	Ash	N/A	ASTM D482 Ignition
	Organochlorine	EPA 3580 dilution	EPA 9020 Microcoulometry
	Viscosity	N/A	ASTM D445 viscometer
Utility water	POHCs	EPA 5030 purge&trap	EPA 8240 GC/MS
	Total chloride	N/A	EPA 300.0 Ion chromatography
	TOX	N/A	EPA 9020 Microcoulometry
	Suspended solids	N/A	EPA 160.1 filter, filtrate evaporation, & gravimetric
	Dissolved solids	N/A	EPA 160.2 filter, residue desiccation, & gravimetric
Quench effluent	POHCs	EPA 5030 purge&trap	EPA 8240 GC/MS
	Total chloride	N/A	EPA 300.0 Ion chromatography

TABLE VI-2. SUMMARY OF ANALYSIS METHODS, CAC INCINERATOR TRIAL BURN, MONSANTO-QUEENY PLANT, ST. LOUIS, MISSOURI (Continued)

Sample	Analytical parameters	Preparation method	Analysis method
	TOX	N/A	EPA 9020 Microcoulometry
	Suspended solids	N/A	EPA 160.1 filter, filtrate evaporation, & gravimetric
	Dissolved solids	N/A	EPA 160.2 filter, residue desiccation, & gravimetric
Scrubber effluent	POHCs	EPA 5030 purge&trap	EPA 8240 GC/MS
	Total chloride	N/A	EPA 300.0 Ion chromatography
	TOX	N/A	EPA 9020 Microcoulometry
	Suspended solids	N/A	EPA 160.1 filter, filtrate evaporation, & gravimetric
	Dissolved solids	N/A	EPA 160.2 filter, residue desiccation, & gravimetric
Stack gas	POHCs	EPA 5040 Thermal desorb	EPA 8240 GC/MS
Stack gas	Particulate	EPA 5 desiccate	EPA 5 gravimetric
	Moisture	N/A	EPA 4 gravimetric
	HCl	N/A	EPA 300.00 Ion chromatography

TABLE VI-2. SUMMARY OF ANALYSIS METHODS, CAC INCINERATOR TRIAL BURN, MONSANTO-QUEENY PLANT, ST. LOUIS, MISSOURI (Continued)

Sample	Analytical parameters	Preparation method	Analysis method
	Temperature	N/A	EPA 2 Thermocouple
	Velocity	N/A	EPA 2 S-Type pitot tube; manometer
Stack gas	CO_2, O_2	N/A	EPA 3 Orsat
Stack gas	O_2	EPA 3A filter, desiccate	EPA 3A continuous ZrO_2 electrochemical
	CO	EPA 10 filter, desiccate	continuous NDIR
Fugitive emissions	VOCs	N/A	EPA 21 FID or PID

N/A = Not applicable
TOX = Total Organic Halides
VOST = Volatile Organic Sampling Train
NDIR = Nondispersive Infrared Spectrometer
GC/MS = Gas Chromatography/Mass Spectroscopy
GFAAS = Graphite Furnace Atomic Absorption Spectroscopy
ICP = Inductively Coupled Plasma Atomic Emission Spectroscopy
FID = Flame Ionization Detector
PID = Photoionization Detector

VI.2 Utility Water

Samples of the utility water line which feeds both the quench and scrubber will be collected from a line tap into precleaned glass bottles every 30 minutes, and then composited for each test run. Final organic composites will be in 40-mL VOA vials, sealed with teflon septa, and no headspace. Analysis for the potential presence of predesignated POHCs will be by purge-and-trap (EPA 5030) and GC/MS (EPA 8240).

VI.3 Quench and Scrubber Effluents

Both quench and scrubber water effluents discharge into open-ended sewer connections, as shown in Figure VI-1. Water samples collected at each location will be by dipper at 30 minute intervals, and subsequent compositing for each test run. Final organic composites will be in 40-mL VOA vials, sealed with teflon septa, and no headspace. Analysis for POHCs will be by purge-and-trap (EPA 5030) and GC/MS (EPA 8240).

VI.4 Particulate and HCl Emissions

The particulate sampling train that will be used in this performance test will meet design specifications established by the Federal EPA. It consists of:

Nozzle - Inconel alloy with sharp, tapered leading edge and accurately measured round opening.

Probe - Glass-lined with a heating system capable of maintaining a minimum gas temperature of 250^{o}F at the exit end during sampling.

Pitot Tube - Type S pitot tube that meets all geometric standards will be attached to the probe to monitor stack gas velocity.

Filter Holder - Pyrex glass with a heating system capable of maintaining a filter temperature of approximately 248^{o}F.

Filter - Glass fiber filter of 3-inch diameter, Whatman Reeve Angel 934 AH.

Draft Gauges - An inclined manometer made by Dwyer with a range of 0 to 10 in. H_2O will be used. A 0 to 1/4-in. H_2O gauge will be used for stack gas velocity pressure measurement.

Impingers - Four impingers connected in series with glass ball joints. The first, third, and fourth impingers will be modified by replacing the impingment tip with a 1/2-inch i.d. glass tube extending to 1/2 inch from the bottom of the flask.

Metering System - Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 5°F, dry gas meter with 2% accuracy, and related equipment to maintain an isokinetic sampling rate and determine sample volume. The dry gas meter is made by Rockwell and the fiber vane pump is made by Gast.

Barometer - Aneroid type to measure atmospheric pressure to within 0.1 in. Hg.

The sampling train, shown schematically in Figure VI-3, will be operated according to EPA Method 5 procedures.

For HCl absorption, impingers 1 and 2 will contain 100 mL of 0.1 N NaOH solution instead of distilled water.

After a particulate/HCl test run has been completed, the sampling train will be carefully moved from the stack test site to the cleanup area. The weight gain of the first three impingers will be measured. Sample fractions will be recoverd as follows:

Container No. 1 - The filter will be removed from its holder, placed in a petri dish, sealed, and labeled.

Container No. 2 - Loose particulate and acetone washings from all sample-exposed surfaces prior to the filter are placed in a glass container that is sealed and labeled. The liquid level is marked after the container is sealed.

Container No. 3 - An unused filter is sealed in a petri dish and labeled as a blank.

Container No. 4 - A minimum of 200 mL of acetone is taken for the blank analysis. The balnk is obtained and treated the same as the acetone washing sample of Container No. 2.

Container No. 5 - NaOH solution from the first and second impingers is placed in a polyethylene container. The impingers and connecting glassware are rinsed with distilled H_2O and the rinse added to the container for shipment. The contents of the third impinger and a distilled water rinse of the impinger are placed in the same container.

Container No. 6 - A minimum of 200 mL of distilled water is taken for the blank analysis. The blank is obtained and treated in a similar manner as the water rinse.

Container No. 7 - A minimum of 200 mL of NaOH solution is taken for blank analysis.

The silica gel from the fourth impinger is weighed and this value recorded on the Sample Recovery and Integrity Sheet with other pertinent data. The silica gel is then discarded.

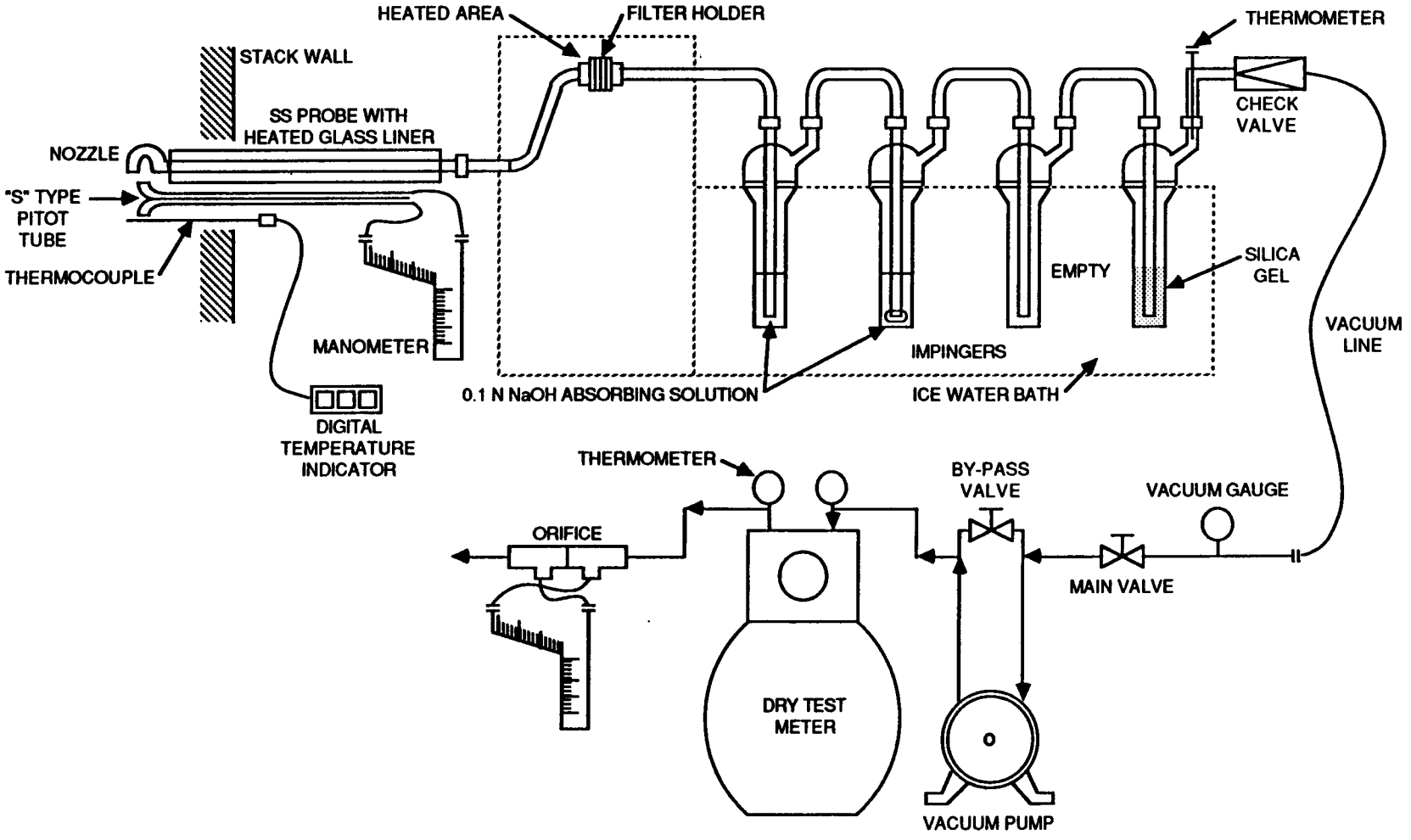


Figure VI-3. Schematic of exhaust gas sampling train for particulate and chloride.

The analytical procedures for particulate determination will follow the methods described in EPA Method 5. Applicable sample containers descibed above will be handled as follows:

Container No. 1 - The filter and any loose particulate matter from this sample container are placed into a tared glass weighing dish, desiccated for 24 hours to a constant weight, and weighed to the nearest 0.1 mg.

Container No. 2 - The acetone washings are transferred to a tared beaker and evaporated to dryness at ambient temperature and pressure, desiccated for 24 hours to a constant weight, and weighed to the nearest 0.1 mg.

Container No. 3 - The blank filter is placed in a tared weighing dish, desiccated for 24 hours to a constant weight and weighed to the nearest 0.1 mg.

Container No. 4 - The acetone blank is treated in an identical manner to the contents of Container No. 2.

The term "constant weight" means a difference of no more than 0.5 mg or one percent of total weight less tare weight, whichever is greater between two consecutive readings, with no less than 6 hours of desiccation between weighings.

The HCl analysis will be conducted according to the procedures outlined in EPA Method 300.0. Sample Container Nos. 5 through 7 will be analyzed separately by ion chromatography. A Dionex ion chromatograph Model No. 10 will be the instrument used in this analysis. The following analytical parameters are expected to be used:

Anion suppressant - 0.025 N H_2SO_4
Anion eluent - 0.003 M $NaHCO_3$, 0.0024 M Na_2CO_3

Ion chromatograph parameters

Flow rate: 23%
"Fast run" anion separation column size: 250 mm bore
Suppressor column: fiber suppressor
Conductivity meter setting: 30 umho full scale
Injection volume: 100 uL

Integrator parameters

Zero set: 10% full scale
Attenuation: 2^6
Chart speed: 0.5 cm/min
Peak width: 0.16
Threshold: 5
Area reject: 500
Quantification: peak area
Run time: 10.0 min
Retention time, chloride: 2:30 +/- 10%

Chloride standard range: 0.20 to 10.0 mg/L

VI.5 POHC Emissions

The two POHCs chosen for this trial burn, ethylene dichloride (b.p. 83°C) and tetrachloroethylene (b.p. 121°C), will be sampled using the Volatile Organic Sampling Train (VOST) and procedures outlined in EPA Method 0030. Analytical procedures will follow that outlined in EPA Methods 5040 (thermal desorption of sorbent tubes) and 8240 (GC/MS for volatile organics).

A safe sampling volume check for Tenax breakthrough of the POHCs shows that the published values of 41 L/2.2 g Tenax for ethylene dichloride (EDC) and 267 L/2.2 g for tetrachloroethylene (Perc) allow ample margin of safety against breakthrough at proper sampling temperatures (68°F).

The calculation of gas sample size needed to demonstrate 99.99% DRE of the POHCs is presented in Table VI-3. Table VI-3 goes one order-of-magnitude beyond to illustrate that sufficient sample volumes can be collected for quantitative analysis.

The sampling train meets the general design specifications of EPA Method 00300 and the VOST Protocol (Protocol for the Collection and Analysis of Volatile POHC's Using VOST, EPA-600/8-84-007). The sampling train is shown in Figure VI-4, and it consists of:

Probe - Stainless steel sheath and glass liner with a heating system capable of maintaining an exit-end gas temperature of 130°C.

Particulate Filter - A plug of glass wool placed in the front of the probe.

Four-way Valve - Stainless steel or teflon four-way valve.

Condensers - Glass coil condensers with water jacket to cool the sample gas stream to 20°C or less before it enters the first sorbent trap.

Sorbent Traps - Borosilicate glass with dimensions of 1.6-cm o.d. by 12.7 cm with both ends necked down to 6.3-mm o.d. Traps are shown in Figure VI-6. The first trap contains a minimum of 1.6 g of Tenax and the second contains a minimum of 1 g of Tenax and 1 g of activated charcoal.

Flask - Borosilicate glass with 250-mL volume and screw cap bored to accept 6.3-mm o.d. tubing. Teflon-backed silicone gaskets are used to make a leak-free seal.

Drying Tube - Teflon container holding approximately 100 g of silica gel.

TABLE VI-3. EXAMPLE CALCULATION OF GAS SAMPLE SIZE NEEDED TO DEMONSTRATE 99.999% DRE

Assume: 1. CAC waste at 1000 lb/hr contains 1.8% EDC and 2.0% Perc as POHCs
2. Stack gas flow rate (Q) is 3400 dscfm (96.3 m^3/min)

$DRE = (W_{in} - W_{out})/W_{in}$

DETERMINE INPUT RATE (W_{in})

EDC W_{in} = (1000 lb/hr)(hr/60 min)(453.6 g/lb)(0.018)

= 136.1 g/min

Perc W_{in} = (1000 lb/hr)(hr/60 min)(453.6 g/lb)(0.02)

= 151.2 g/min

CALCULATE OUTPUT RATE (W_{out})

Assume 99.999% DRE as target for sensitivity

Then, $0.99999 = (W_{in} - W_{out})/W_{in}$

EDC W_{out} = 0.00136 g/min = 1360 ug/min

Perc W_{out} = 0.00151 g/min = 1510 ug/min

STACK GAS CONCENTRATION

W_{out}/Q = concentration, ug/m^3

Perc = (1510 ug/min)/(96.3 m^3/min) = 15.7 ug/m^3

EDC = (1360 ug/min)/(96.3 m^3/min) = 14.1 ug/m^3

Need >10 ug/m^3 (ng/L) for efficient quantitation

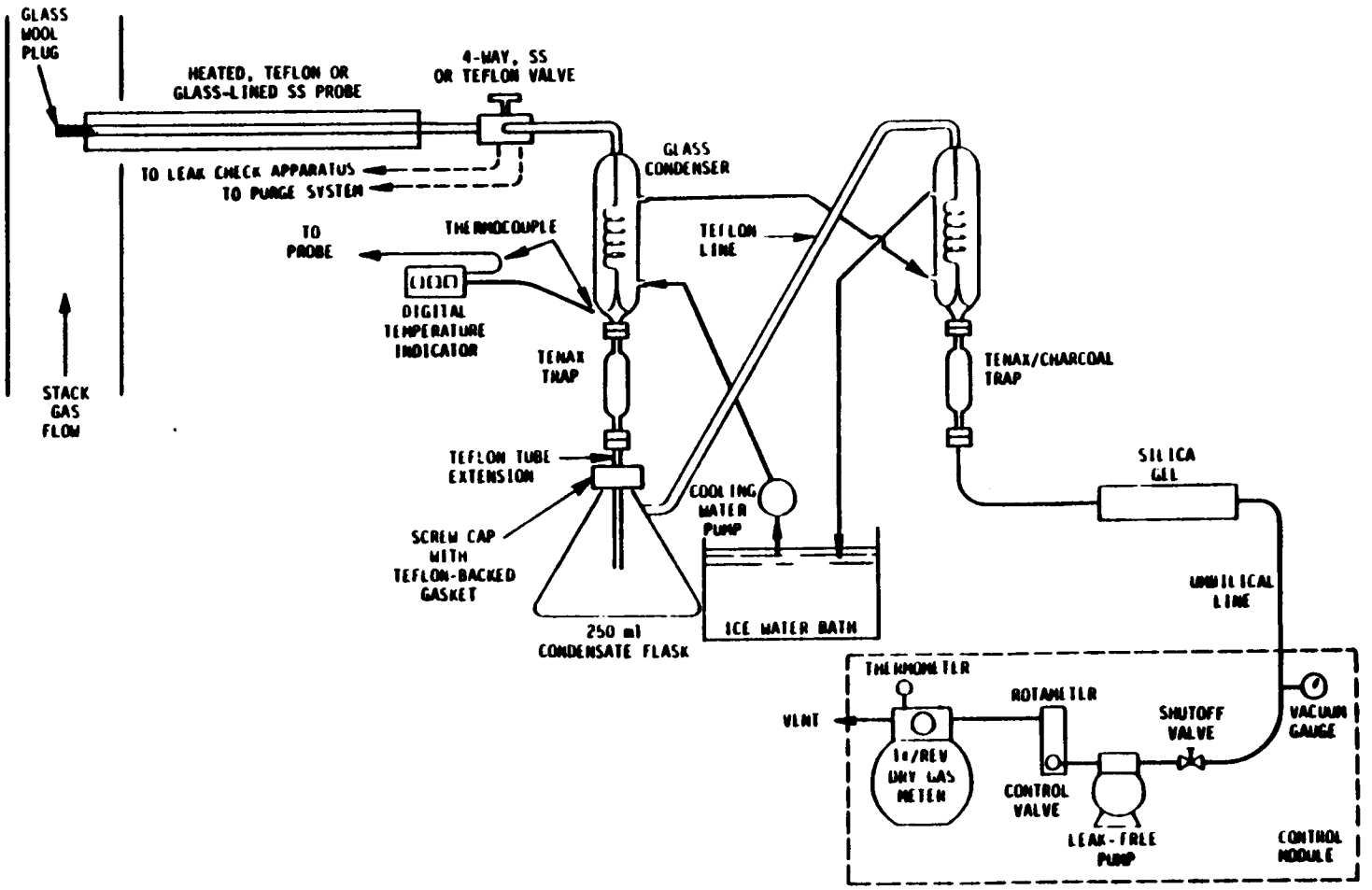


Figure VI-4. Schematic of volatile organic sampling train (VOST).

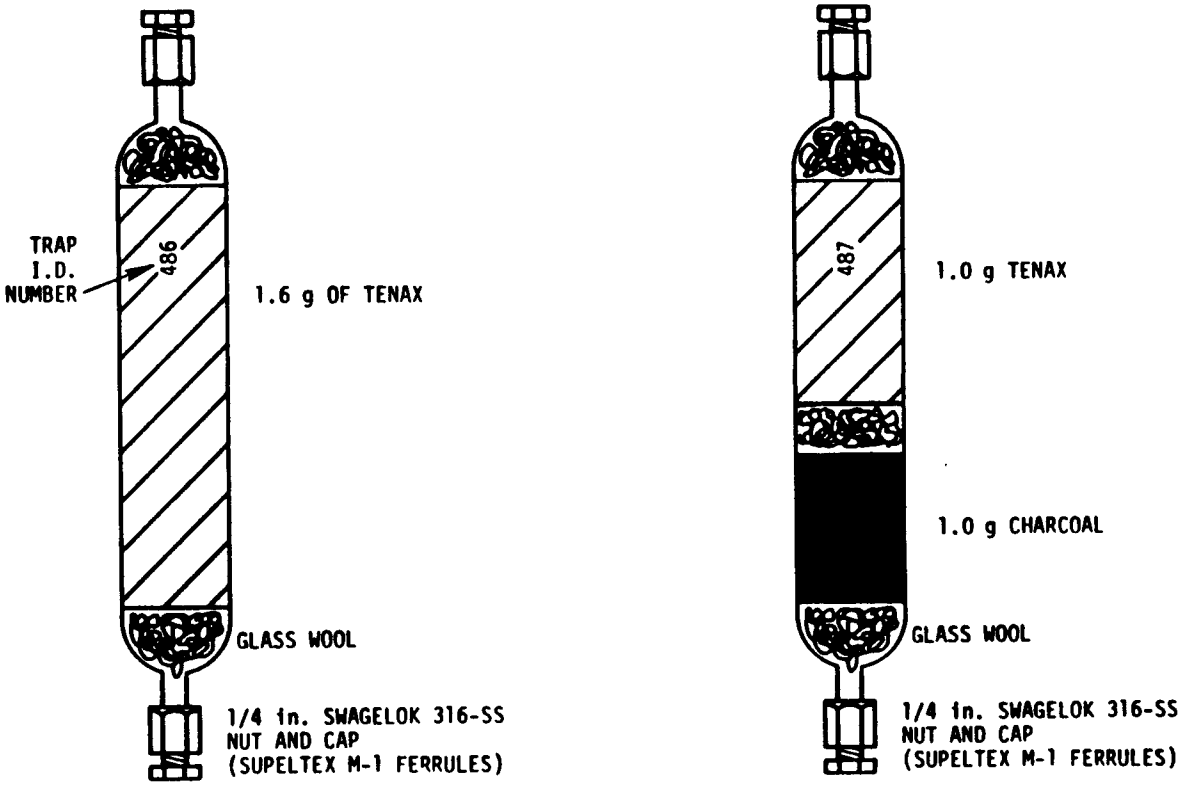


Figure VI-5. Sorbent trap configurations.

Metering System - Vacuum gauges, leak-free diaphragm pump, calibrated rotameter, Singer dry gas meter measuring 1 Liter/revolution within 2% accuracy for flow rates between 0.25 and 1.0 L/min.

The trap blank check apparatus consists of:

Thermal Desorption Unit - Modified Supelco high-capacity gas purifier oven. A Variac is used to control oven temperature and a thermocouple is used to monitor temperature.

Purge and Trap Unit - Tekmar Model LSC-2 with all teflon transfer lines replaced with 1.6-mm o.d. stainless steel tubing. The analytical trap consists of a 15-cm long section of Tenax, 3.7-cm section of silica gel, and a 3.7-cm section of charcoal.

Analyzer - Perkin Elmer Model 990 gas chromatograph with a flame ionization detector (GC/FID). The GC column is a 6 ft x 1/8 inch o.d. stainless steel column packed with 1% SP-1000 on Carbopak B.

The analytical apparatus system to be used in these tests was assembled by PEI personnel in accordance with EPA Methods 5040 and 8240 and the VOST Protocol. It consists of:

Thermal Desorption Unit - Modified Supelco high-capacity, gas-purifier oven ("clamshell" oven). A Variac is used to control oven temperature, and a thermocouple is used to monitor the temperature.

Purge and Trap Unit - Tekmar Model LSC-2 with all teflon transfer lines replaced with 1.6-mm o.d. stainless steel tubing. The analytical trap consists of a 15-cm long section of Tenax, 3.7-cm section of silica gel, and a 3.7-cm section of charcoal.

GC/MS System - Finnigan model 4023 with quadrupole mass spectrometer. The separation column is 1.848-m by 2-mm i.d. glass, packed with 1% SP-1000 on Carbopak B (60/80 mesh). The mass spectrometer scans from 35 to 335 m/e when gaseous standards are used. The GC/MS interface is an all-glass jet separator. The data acquisition and processing system that controls the mass spectrometer consists of a Data General Nova 3 computer with Perkin Elmer/Wangco 10 mega-byte dual disk drive running Finnigan Incos software.

Reagent and Materials Preparation -- Each of the reagents, aparatus, parts, and standards have a procedure or specification to meet for high quality measurement results. The VOST reagents and materials cosist of:

Tenax - 2,6-Diphenylene oxide polymer (35/60 mesh) Soxhlet extracted for 24 hours with glass-distilled methanol (Burdick and Jackson pesticide residue grade or equivalent). After extraction, the Tenax is transferred to a clean ceramic evaporating dish and dried for 4 hours under an infrared lamp. The Tenax and evaporating dish are then placed in a vacuum oven at 50°C and 20 in. Hg vacuum for 6 hours to complete drying. Finally, the Tenax is transferred to a clean amber glass bottle with a teflon-lined screw cap and placed in a glass aquarium containing activated charcoal.

Charcoal - Petroleum-based charcoal (SKC Lot 208, calgon-Type GW20X50, or equivalent) is prepared by heating to 190°C in a vacuum oven under a slow nitrogen purge according to the following procedures:

1) Charcoal is placed in a cylindrical metal container opened at the top and connected to a source of charcoal-filtered nitrogen at the bottom.

2) Nitrogen purge flow is set at 50 mL/min.

3) The charcoal and metal container are placed in a vacuum oven set at 190°C and approximately 5 in. Hg vacuum for 6 hours.

Glassware and Teflon Parts - All glass parts (traps, culture tubes, flasks, and condensers), teflon fittings, and sample-exposed connecting links are cleaned with a nonionic detergent (Alcojet) and rinsed thoroughly with charcoal-filtered deionized water. The parts are then oven dried at 110°C for 8 hours. All parts are then wrapped in aluminum foil and stored in sealed glass aquariums containing activated charcoal.

Metal Parts - Sorbent trap end-plugs and stainless steel unions are ultrasonically cleaned for 15 minutes in a hot nonionic detergent solution, then rinsed with charcoal-filtered deionized water, air-dried, and finally heated in a muffle furnace for 2 hours at 400°C. Cleaned parts are stored in amber bottles placed in sealed glass aquariums containing activated charcoal.

Glass Wool - Pyrex wool filtering fiber is Soxhlet extracted for 16 hours with glass-distilled methanol, placed in a clean ceramic evaporating dish, and dried under an infrared lamp in an exhaust hood for 4 hours. The glass wool and dish are then placed in a vacuum oven and heated at 110°C and 20 in. Hg vacuum for 6 hours. The cleaned glass wool is stored in amber glass bottles with teflon-lined screw caps until use.

Water - Charcoal-filtered, deionized water is used for trap leak checking prior to blank analysis. Water used for

analytical steps is further treated by boiling for 15 minutes.

Nitrogen - For purging sorbent traps during thermal conditioning, nitrogen is passed through an inline gas purifier containing 5A° molecular sieve and activated charcoal. The charcoal bed is replaced with each new nitrogen cylinder.

Analytical Trap - the analytical trap consists of the following components: Tenax (60/80 mesh), chromatographic grade or equivalent; silica gel, Davison Chemical (35/CU mesh), Grade 15 or equivalent; charcoal, petroleum-based (SKC Lot 104 or 208, Calgon Type GW20X50, or equivalent).

Stock Standard Solution - The stock standard solutions are prepared weekly from pure standard material by diluting with glass-distilled methanol.

Bromofluorobenzene (BFB) Tuning Check Standard - A solution of 50 ug/mL BFB in methanol is prepared and 1 uL of this standard is injected on the column each day for the MS tuning check.

Internal Standard - A known concentration of d_6-benzene and hexafluorobenzene is prepared in methanol, such that a 4-uL on-trap injection produces an amount on column in the range of the amount of the target compounds.

Assembly and Conditioning of VOST Sorbent Traps -- Sorbents are loaded into the glass tube by use of a modified rifle shell loader. Tenax tubes are loaded with 1.6 grams of Tenax, and Tenax/charcoal tubes are loaded with 1.0 grams of each sorbent. A small section of teflon tubing is used to help transfer the sorbent from the loader into the sorbent tube. Sorbent beds are held in place by glass wool plugas. Sorbent tube ends are sealed with a Swagelok stainless steel cap and Supeltex M-1 ferrule. Each sorbent trap is stored in a clean glass culture tube with a teflon-lined screw cap. All culture tubes are stored in sealed glass aquariums containing activated charcoal.

Once assembled, the sorbent traps are conditioned by passing nitrogen (30 mL/min) through the trap for 28 hours. Traps are heated in an oven at 190°C during conditioning. After conditioning the traps are returned to a culture tube and stored in a friction-top metal container, which also contains activated charcoal. Both the culture tubes and the metal can are purged with nitrogen to remove air contaminants before the traps are placed into them.

Blank Checking Procedure -- each set of traps is blank checked to ensure that the tubes contained less than 5 ng of the target compounds or any other contaminant. The target compounds include the designated POHCs and acetone, benzene, and hexane.

The following procedures are used to blank check each pair of sorbent traps:

1) A pair of traps is connected with the charcoal side of the Tenax/charcoal tube connected to the inlet carrier gas. The traps are connected by stainless steel unions.
2) With the outlet of the paired traps (Tenax tube sampling inlet) still capped, the system is leak-checked at approximately 30 psig, either by immersing the traps in a pan of distilled water and observing the water for bubbles or by checking around the fitting with a thermal conductivity gas leak detector (e.g., GOW-MAC^{R}). Any leaks are corrected.
3) The outlet of the paired traps is connected to the Tekmar purge and trap system. The traps are then placed in the oven and desorbed for 10 minutes.
4) After desorption, the sample collected in the purge and trap apparatus is injected into the GC.
5) To minimize contamination, carrier gas flow is maintained at all times and traps are disconnected by the following procedure:
    a) After cooling, the outlet of the paired traps is capped.
    b) The union between the traps is disconnected and both ends are capped.
    c) The outlet of the Tenax/charcoal tube is disconnected and capped.
6) Traps are immediately placed back in their respective culture tubes, which are taped together to keep the paired traps as a unit.

Sample Collection Procedure. -- The VOST is assembled at the sampling site. During sample collection, the end caps of the sorbent traps are placed back into the culture tubes in which the traps were stored. VOST operation procedures follow that prescribed in EPA Method 0030.

After sampling, the traps are disconnected, capped, placed in the culture tubes, sealed, and labeled. All traps are placed placed in the culture tubes with point of entry nearest the screw cap. The culture tubes are then purged with charcoal-filtered nitrogen and sealed. The culture tubes are placed back into the metal cans, the metal can is purged with nitrogen prior to sealing at the end of the test day. All samples are transported in an ice chilled cooler and refrigerated in the laboratory until analysis

Analytical Procedure -- The VOST sorbent tubes are analyzed according to the VOST Protocol. A schematic of the desorption and analysis system is shown in Figure VI-6. All target compounds are quantified using the inetrnal standard method with d_6-benzene as an internal standard added to all samples and external calibration standards. calibration standards are loaded onto clean sorbent traps via the flash vaporization method in EPA Method 0030.

A response factor (RF) is calculated for each compound versus its internal standard by the following equation:

$$RF = A_s/C_{is}/A_{is}C_s$$

where A_s = area of the characteristic ion for the analyte to be measured

A_{is} = area for the characteristic ion of the internal standard

C_{is} = amount (ng) of the internal standard

A_s = amount (ng) of the analyte in the calibration standard

The RF's are used to compare initial calibration data with daily check standards. A computer generated least squares fit to a linear or quadratic equation ($A_s/C_{is}/A_{is}$ vs. C_s) is used to produce a calibration curve for each analyte. A minimum five-point plus blank curve is generated weekly. The calibration standards are run from low to high level to minimize the effect of carryover of residual compounds from one standard to the next.

VI.6 CO and O_2 Stack Gas Concentrations

Carbon monoxide (CO) and oxygen (O_2) concentrations will be measured continuously through the test run periods. The sampling procedures are those of EPA Method 10 for CO and EPA Method 3A for O_2. A schematic of the sampling system is shown in Figure VI-7.

The CEM sampling system consists of an inconel probe with an in-stack particulate filter, a three-way ball valve at the probe exit for calibration gas introduction, a stainless steel condenser for moisture removal, a teflon sampling line, and a teflon diaphragm pump to supply the analyzer sampling manifold. The CO analyzer is an NDIR detector; the O_2 analyzer is an electrochemical cell detector. Both analyzers will be calibrated at the beginning and end of each test run with three gas standards in the analytical range and zero nitrogen. Span and zero checks will be conducted at the midpoint of each test run. Calibration data will be reduced by means of a linear regression analysis, and the linear equation will be used to quantitate stack gas concentrations. The output from the analyzers will be recorded continuously on analog-type strip chart recorders for a permanent record.

A Bendix Model 8501-5CA continuous nondispersive infrared (NDIR) analyzer will be used to measure CO. It has concentration ranges from 0-50 ppm, 0-250 ppm, 0-500 ppm, and 0-1000 ppm, with a minimum detection limit of 0.5 ppm in the 0 to 50 range. It has linearity of 1% of span, precision of 1% of span, noise at

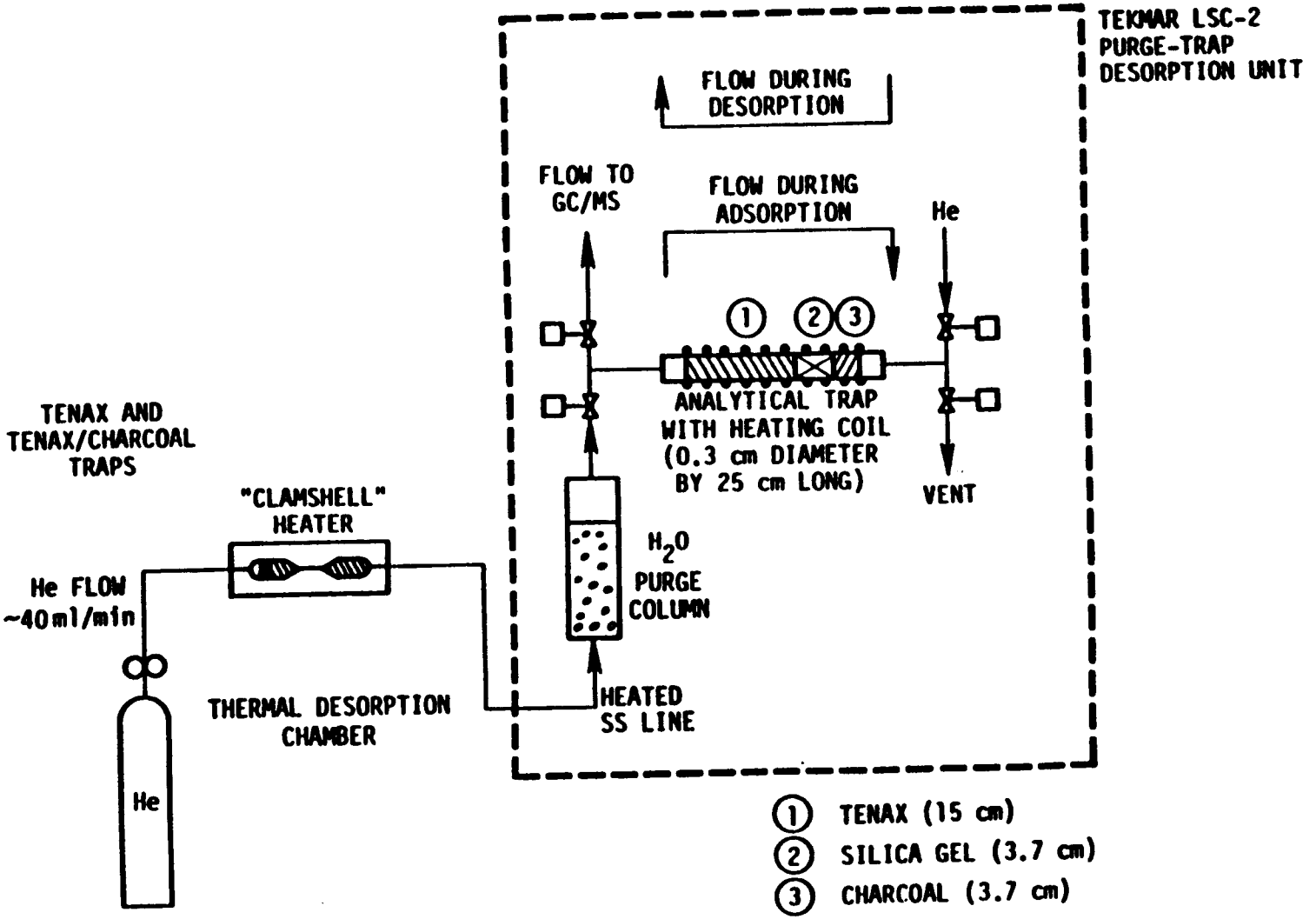


Figure VI-6. Schematic of typical VOST sorbent trap desorption and purge and trap apparatus.

0.5% of span, zero drift stability of 1% for 24 hr, span drift stability of 1% for 24 hr, electronic response time of 0.7 seconds to 90% of scale, CO_2 interference ratio of 40,000 to 1, and H_2O interference ratio of 20,000 to 1.

A Data Test Corporation Model 303 zirconia cell detector will be used to measure oxygen. The analyzer is operated in the 0 to 25 percent range and will be calibrated with zero nitrogen and gas standards of 4, 7, 15, and 21 percent oxygen.

VI. Fugitive Emissions

Fugitive emissions screening for leaks will be conducted using EPA Method 21 for VOC screening. The sources with potential to leak will be tagged first for identification, and then source screened from the CAC waste feed pump to the scrubber/absorber tower.

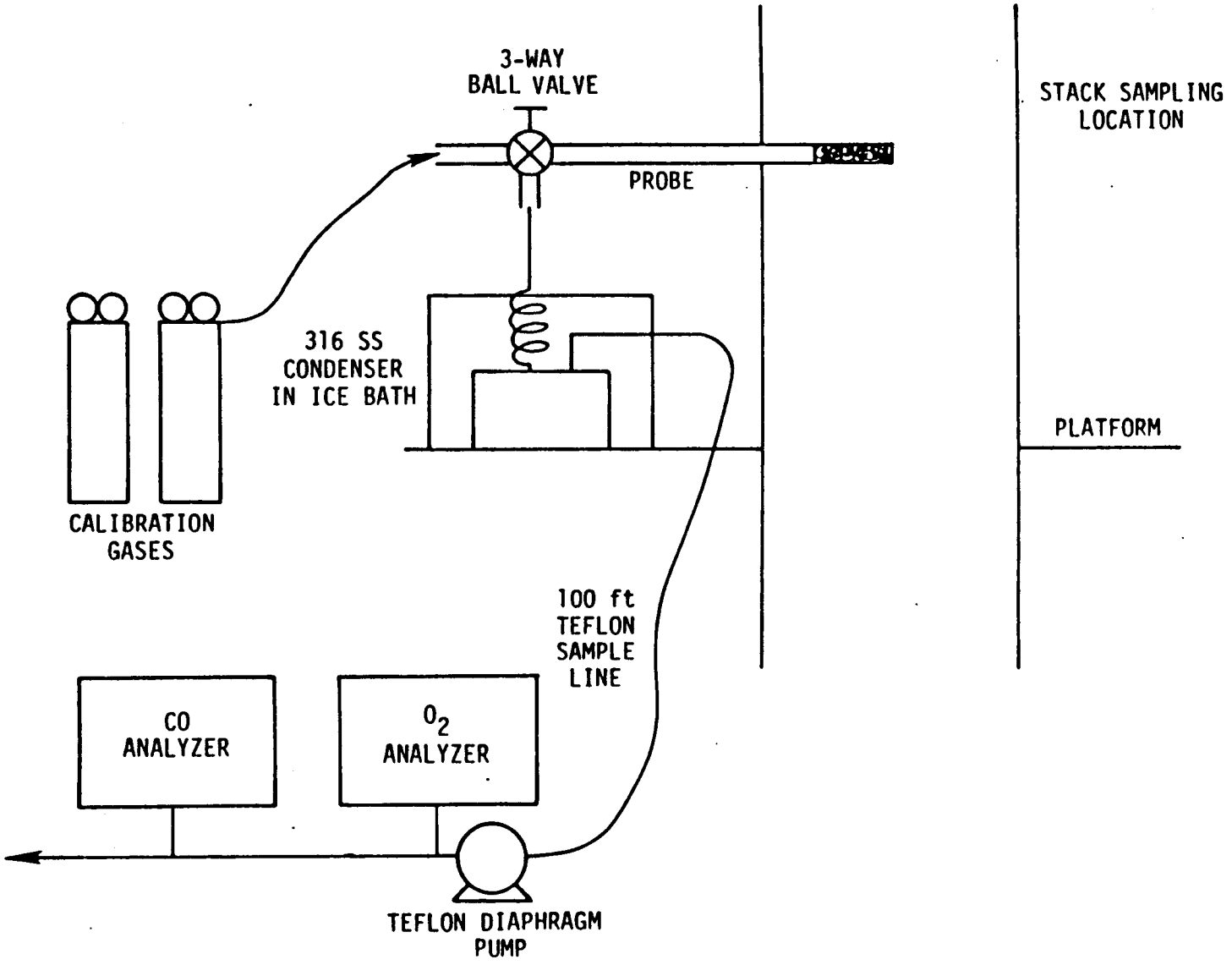


Figure VI-7. CEM sampling system for CO and O_2.

VII. QUALITY ASSURANCE/QUALITY CONTROL PLAN

VII.1 Introduction

The purpose of this QA/QC Plan is to provide the environmental measurement systems employed in the trial burn with the procedures and documentation which demonstrate that the measurement has a defined accuracy and precision associated with it. Described herein will be procedures for maintaining instruments and equipment in a state of calibration (defines the accuracy or bias error), procedures for measuring/calculating the repeatability of a measurement (defines precision, or random error), procedures for maintaining a state of cleanliness (eliminates interferences or contamination), and the paper trail which documents that the methods are performed to instructions, calibrated, within method performance standards, traceable to NBS standard reference materials, audited, and samples were secure from tampering.

VII. Project Description

Section V of this trial burn plan describes the trial burn protocol. Table V-2 summarized the test conditions to be employed during the trial burn. Section VI described the sampling and analysis plan for the trial burn testing. Tables VI-1 and VI-2 summarized the sampling and analytical programs of the CAC incinerator trial burn.

The QA/QC measures to be employed in this trial burn test program are summarized in Table VII-1, which describes the number of additional samples required for analysis beyond those collected in the sampling program.

VII.3 Project Organization and Responsibility

The sampling and analytical subcontractor, PEI Associates, Inc., has developed and implemented a corporate-wide quality assurance program. This program confirms their policy to provide high quality technical studies and directs project managers to to conform to plans which will assure data quality and corrective action when necessary. This corporate program is, in turn, supported by specific technical area quality assurance plans.

Figure VII-1 presents the PEI Quality Assurance organization chart and illustrates the relationship of the Emission Measurement Group's and Laboratory Group's quality assurance activities to the total corporate quality assurance effort.

Responsibility for the PEI Corporate Quality Assurance Group rests with Mr. Charles E. Zimmer, Senior Vice president, with specialists assigned to each operating/functional area. In his position as a member of top management of the company, Mr. Zimmer has the authority to require that each Project manager gives evidence of compliance with QA Project plans from the initial

TABLE VII-1. QUALITY ASSURANCE MEASURES FOR THE TRIAL BURN

Parameter	Total number of samples per three test conditions	Quality control samples to determine accuracy and precision			
		Blanks	Duplicates	Matrix spikes	Reference standards
Liquid waste	9				
POHCs	9	MeOH	2	1	1[a]
TOX	9	NA	1	NA	1[a]
Metals	9	HNO_3	1	1	1[a]
Btu	9	NA	2	NA	NA
Moisture	9	NA	0	NA	NA
Ash	9	NA	2	NA	NA
Viscosity	9	NA	1	NA	NA
Aqueous streams	27				
POHCs	27	NA[b]	3	1	1[a]
Chloride	27	NA[b]	3	1	1[a]
TOX	27	NA[b]	1	NA	NA
TSS	27	NA	1	NA	NA
TDS	27	NA	1	NA	NA
Stack gas					
POHCs	27 pairs of VOST tubes	3-field 2-trip 2-lab	NA	NA	1[a]
	9 condensates	NA	1[c]	NA	1[a]
Particulate	9	1-filter 1-acetone	NA	NA	1[d]
HCl	9	1 Na_2CO_3	1	1	1[a]
CO, O_2	continuous	zero gas	NA	NA	cal gases
Fugitives	one screen survey	zero gas	NA	NA	cal gas

[a] Independently prepared control sample to verify instrument calibration.

[b] Utility water acts as blank for quench and scrubber effluent.

[c] If sufficient condensate is collected.

[d] Participation in EPA performance audit programs for EPA Methods 3 and 5.

planning stages through data collection, validation and analysis, to the delivery of the final report to the client.

Administratively, the QA specialists report to the Senior Vice President and are not subject to the line authority of the managers of the groups or projects to which they are assigned. However, to allow efficient flow of QA information and reports, and to initiate corrective actions, the QA specialists functionally report to the division directors.

Figure VII-2 presents the administrative organizational structure of the Emission Measurement Group. Their function is the collection and assessment of stationary source emission data. Figure VII-3 presents the administrative organizational structure of the Laboratory. Their function is the analysis of environmental samples. Both groups must meet the objectives for which the project is being conducted. Mr. Tom Wagner, as Quality Assurance Coordinator, has the overall responsibility for assessing the implementation of the QA project plan. The Project Manager, Mr. Dale Hershey, has the responsibility to implement project design and the QA project plan. Figure VII-4 illustrates the detailed functional organizational chart showing the quality control loops which allow assessment of each function in generating the data and provide for corrective feedback.

VII.4 QA Objectives for Measurement Data

Table VII-2 presents the methods that will be used to obtain the critical data of the trial burn performance testing and the corresponding objectives for precision, accuracy, and completeness. Other parameters will be measured in the test program but do not warrant setting of QA objectives.

VII.5 Sampling and Analysis Procedures

All sampling and analysis procedures have been described in Section VI of this trial burn plan. The field and laboratory data sheets used to document the proper implementation of a method are given in Appendix E.

VII.6 Sample Custody

Samples are recovered from the sampling systems after each test run. sample recovery is carried out in a suitable area sheltered from wind, dust, or other contaminants to preserve sample integrity. Sample recovery procedures will follow that outlined in detail in each applicable method used.

All sample containers are identified using the appropriate section of the three-part label, shown in Figure VII-5. One section of the sample is attached to the field test data sheet for positive identification purposes. After recovery, all sample containers are sealed. The liquid level height is marked to indicate possible samle loss during shipment or holding. Data

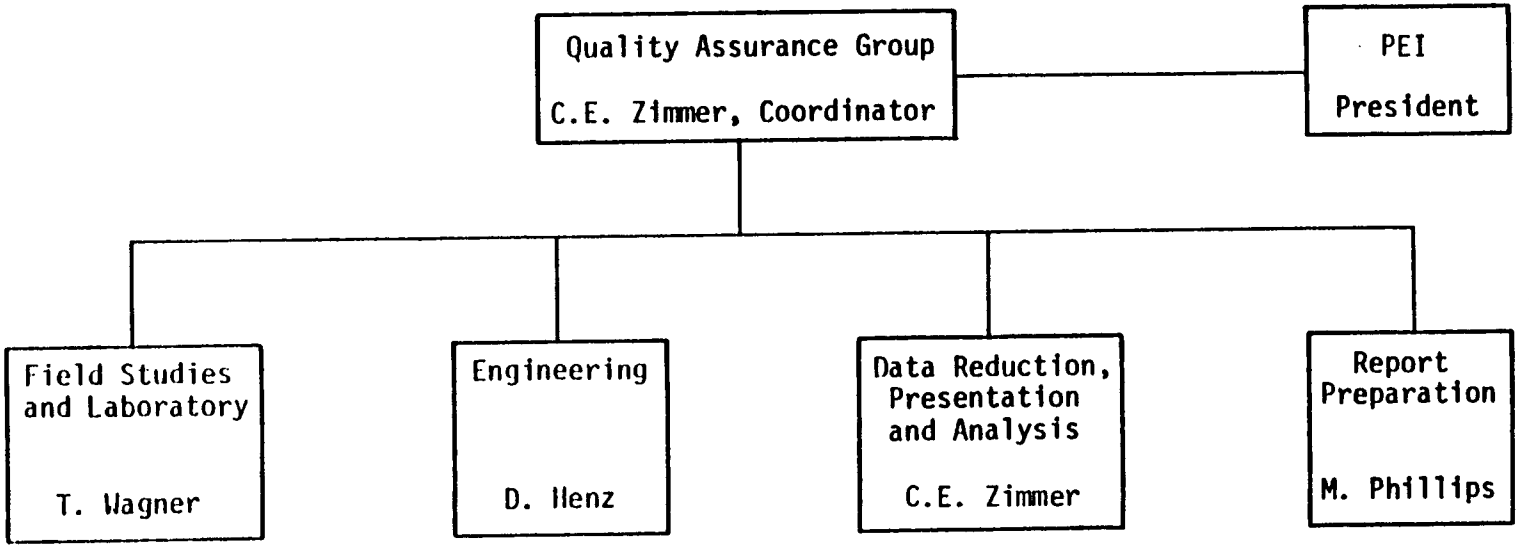


Figure VII-1. Organization of PEI corporate quality assurance.

TABLE VII-2. QUALITY ASSURANCE OBJECTIVES FOR CAC INCINERATOR TRIAL BURN MEASUREMENT DATA

Parameter	Method	Expected results, %		
		Precision	Accuracy	Completeness
Liquid waste POHCs	EPA 8240 (GC/MS)	+/-30[a]	+/-50[a]	90
Aqueous samples POHCs	EPA 8240 (GC/MS)	+/-30[a]	+/-50[a]	90
Stack gas POHC conc.	EPA 0030 (VOST)	+/-25[b]	+/-50[b]	90
Velocity/flow	EPA 2	+/-5[c]	+/-10[d]	90
CO_2, O_2	EPA 3	+/-0.5[e]	+/-0.5[e]	90
Particulate	EPA 5	+/-10[c]	not listed	90
HCl conc.	EPA 5; EPA 300.0	+/-30[a]	+/-10[f]	90
Oxygen conc.	EPA 3A	NA	+/-1[g]	90
CO conc.	EPA 10	NA	+/-10[g]	90

[a] Based on limits in EPA-600/8-84-002.

[b] Based on limits in the VOST Protocol, EPA-600/8-84-007.

[c] Based on method collaborative study, EPA-600/4-76-014.

[d] Estimated.

[e] As listed in EPA QA Handbook, Vol. III, EPA-600/4-77-027b.

[f] Limit for analysis of control samples.

[g] Based on minimum performance specifications in 40 CFR 60, Appendix B, for CEMS.

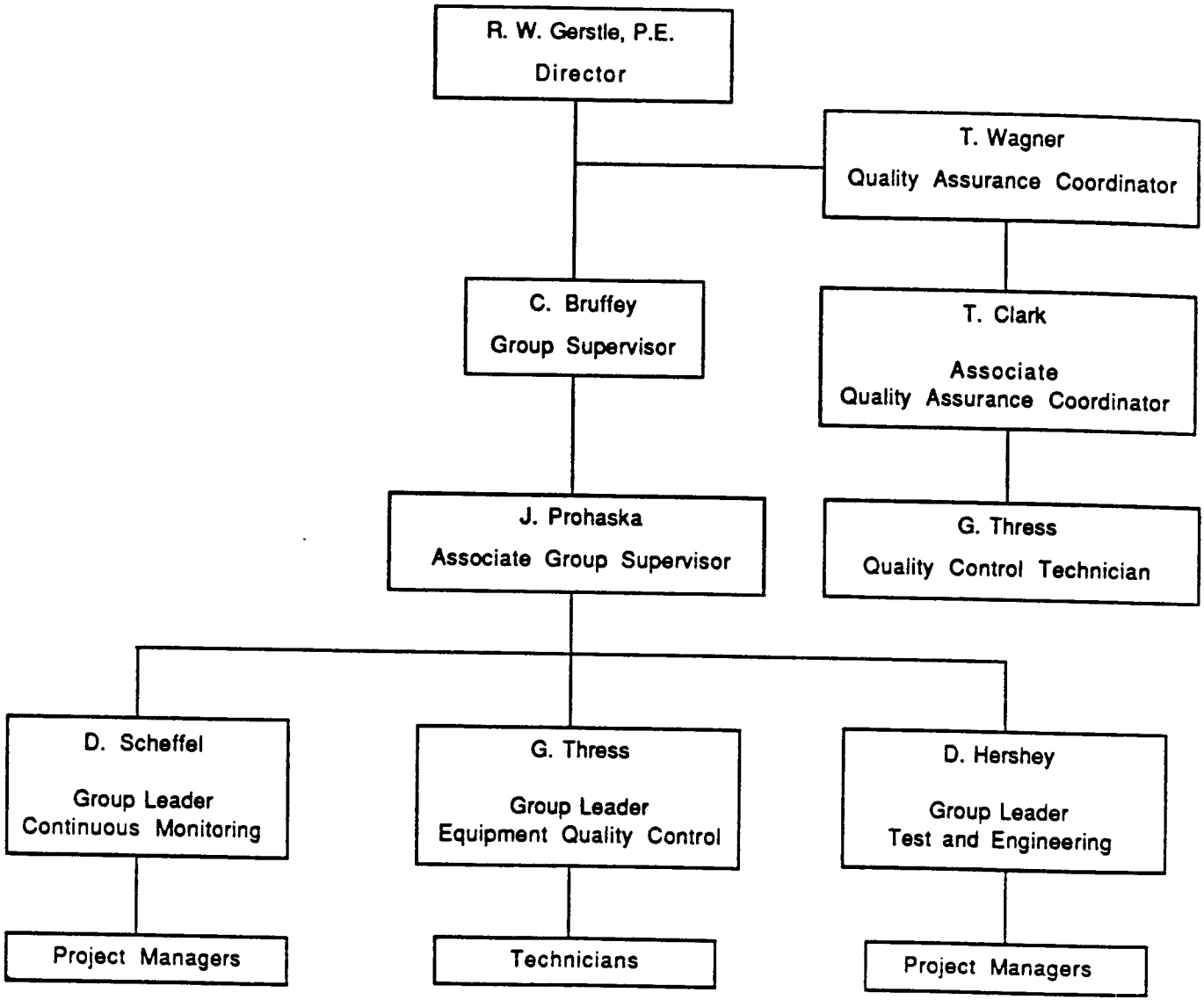


Figure VII-2. Emission measurement group organization chart.

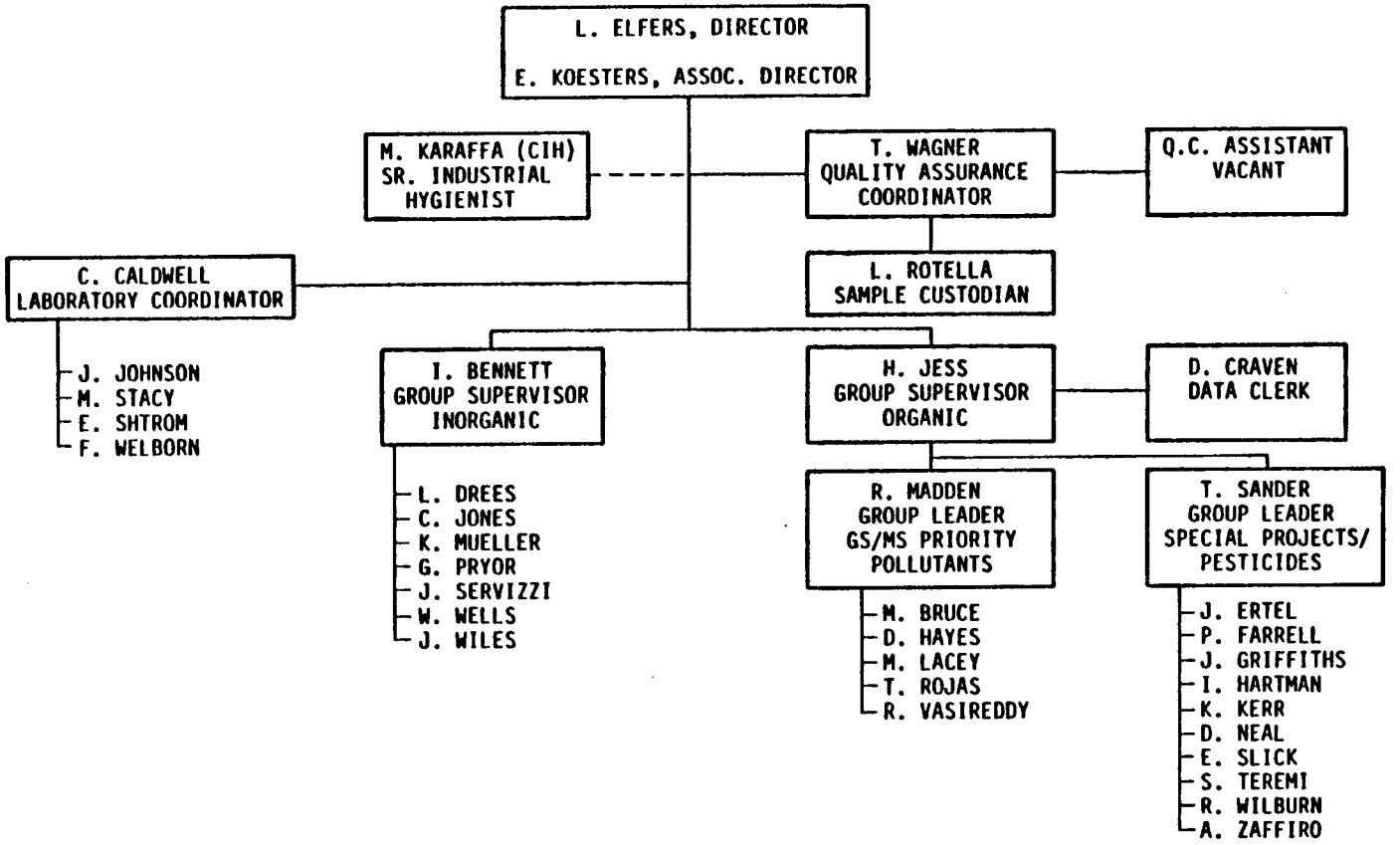


Figure VII-3. Laboratory organization chart.

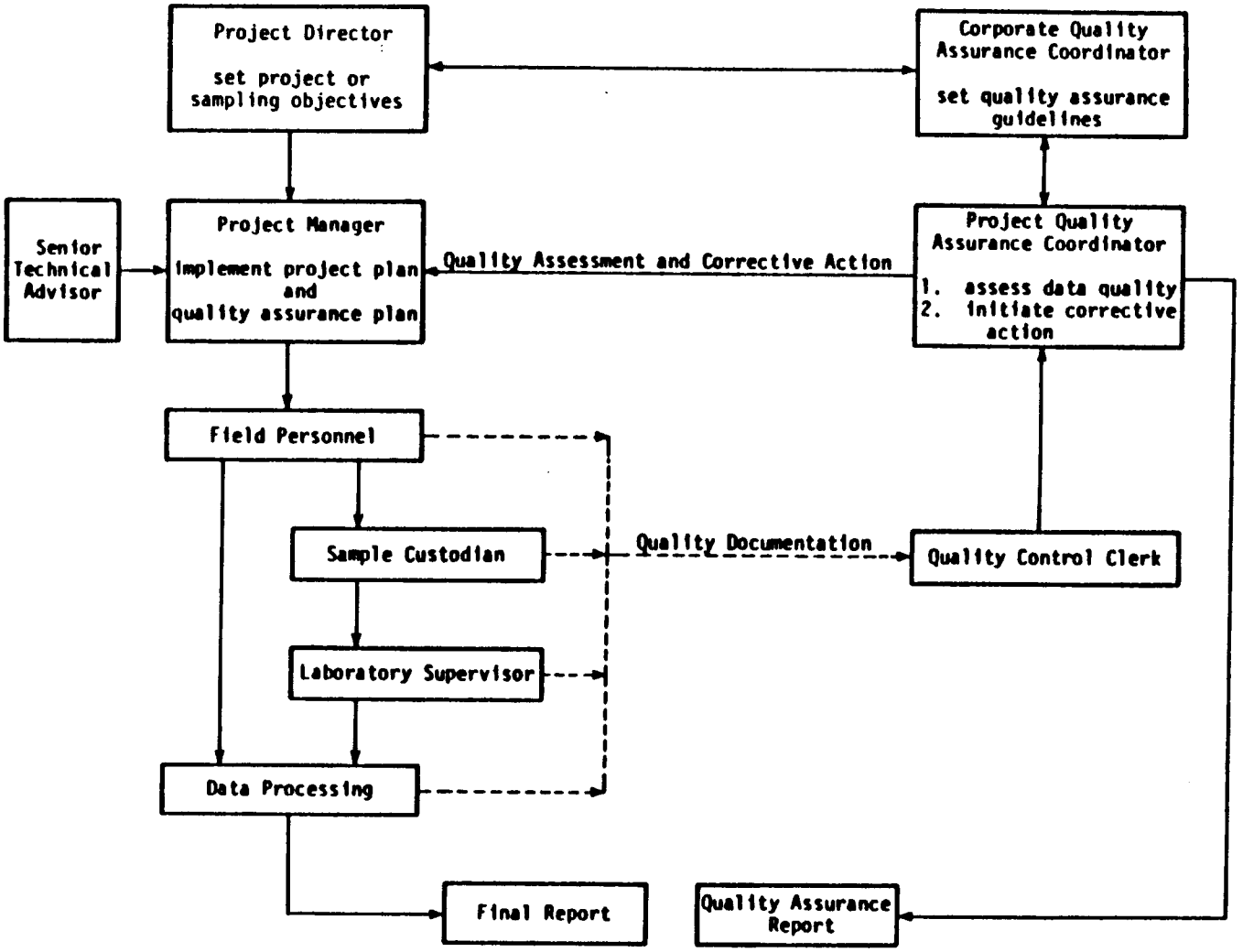


Figure VII-4. Project responsibilities and quality assurance loops.

relative to samples, collected during each test, are recorded on a sample recovery and integrity data sheet (see Appendix E). Samples, along with the sample recovery and integrity data sheets, are under the custody of the Project Manager until custody is transferred when samples are turned over to the laboratory. Sample storage boxes with padlocks are used to store and transport samples. Chain-of-custody is recorded on the sample integrity sheets.

Custody procedures in the laboratory follow the requirements of the U.S. EPA National Enforcement Investigation Center (NEIC) in Denver, Colorado. They include a computerized daily sample log, analysis requisition form, sample control record, laboratory data record, and procedures for managing raw data, calibration curves and charts.

VII.7 Calibration Procedures and Frequency

All manual stack gas sampling equipment will be calibrated before the test program according to procedures outlined in Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, EPA-600/4-77-027b. Onsite calibration checks also will be made prior to the start of testing to preclude the possibility of damage from packing and transport. Table VII-3 summarizes the stack gas sampling equipment calibrations to be performed in preparation for this project. The dry gas meter boxes are also calibrated after the trial burn. Some of the calibration data forms which will be used are presented in Appendix E.

The continuous emission monitoring systems (CEMS) will be calibrated initially to establish linear regression coefficients, calibration error, minimum detectable limit, specification for response time, and sampling system bias. Daily CEMS calibrations establish linear regression curves and drift error. Copies of the initial and daily CEMS calibration and performance evaluation forms are given in Appendix E.

For POHC analysis, the GC/MS will be calibrated weekly by using a minimum of a zero point plus four upscale points. The calibration curve is generated for each target POHC by the internal standard technique. In addition, daily calibration checks are made for each target POHC. The daily check standard must be within 15% (except for the lowest point) of the weekly calibration or the instrument is recalibrate. A GC/MS mass calibration and tune check is conducted daily prior to any other calibration or analysis. QA documentation will follow the forms provided in EPA-SW-846 for the above activities.

For POHC analysis, the standards will be prepared solutions in methanol. The internal standard will be d_6-benzene. For VOST analysis, the calibration standards and internal standard spike will be flash vaporized onto a pair of sorbent traps and analyzed in the same manner as the sample traps. The daily mass assignment calibration check will be conducted with

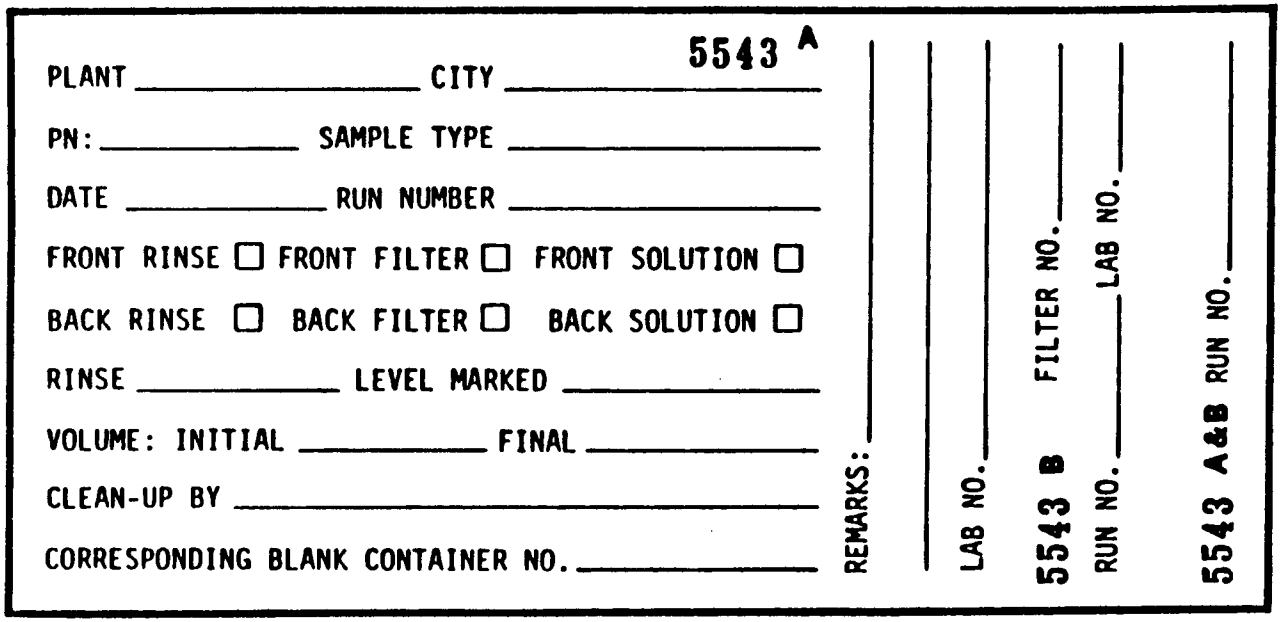


Figure VII-5. Three part sample label.

perfluorotributylamine (FC-43), and the daily tune check will be conducted with bromofluorobenzene (BFB).

For chloride analysis, the ion chromatograph is calibrated for each analysis run on a set of samples, and a reference solution is run after every tenth sample. If the reference solution analysis is not within +/-10%, the instrument is recalibrated and the previous ten samples are reanalyzed.

For particulate analysis, the analytical balance is checked each time filters are weighed.

For metals analysis, a minimum of one sample per ten is spiked with an element of interest and the recovery efficiency determined. Typically, recoveries must remain within +/-20% of theoretical before instrument recalibration is required.

VII.8 Data Reduction, Validation and Reporting

Data reduction and reporting have been shown to be great potential sources of system error. Most of PEI's test method calculations and analytical calculations are performed by a validated computer program to minimize error. The sampling field data sheets are set up on a standard computer card to allow accurate input of data into the computer by individuals unfamiliar with testing procedures. The data printout is then validated by comparison with the field and analytical data sheets. Hand calculation check are also made to validate the computer output.

All data generated by each phase of a laboratory or field sampling program are reviewed by a group leader or group supervisor. This involves verifying that the appropriate method was used, the detection limit is appropriate, the proper number of significant figures are reported, and the data were calculated properly (hand calculation check). The data must be reviewed by the Quality Assurance Coordinator before releasing the data for report preparation.

VII.9 Internal Quality Control Checks

The internal quality control checks for the parameters listed in Table VII-3 follow the procedures contained in Volume III of the QA Handbook and the VOST Protocol. Table VII-4 lists the additional calibration checks that will be performed on the sampling equipment on site just prior to the testing to ensure that the equipment was not damaged during transport.

Where available, only analytical reagents which conform to the specifications of the method are used. All reagents entering the laboratory are labeled, and are entered on the Reagent Procurement Record. Solutions of reagents are labeled when prepared with proper nomenclature, concentration, date prepared, and initials of the preparer. Class A volumetric glassware only

TABLE VII-3. FIELD EQUIPMENT CALIBRATION SUMMARY[a]

Equipment	Calibrated against	Allowable error
Method 5 meter box	Wet-test meter	Y ±0.02 Y ΔH @ ±0.15 post-test Y ±0.05 Y
VOST meter box	Bubble meter	Y ±0.02 Y[b] post-test[b] Y ±0.05 Y[b]
Orsat	Certified cylinder gas	±0.5%
Pitot tube	Geometric specifications	See EPA Method 2
Digital indicator	Millivolt signal	±0.5%
Thermocouple	ASTM-3F thermometer	±1.5%
Impinger (or condenser thermometer	ASTM-3F	±2°F
Dry gas thermometer	ASTM-3F	±5°F
Balance	Type S weights	±0.5 g
Probe nozzles	Caliper	±0.004 in.
Barometer	NBS traceable barometer	±0.1 in.Hg

[a] As recommended in Quality Assurance Handbook for Air Pollution Measurement Systems: Volume III. Stationary Source Specific Methods. EPA-600/4-77-027b, August 1977.

[b] As recommended in Validation of the VOST Protocol - Field Validation Phase. EPA Contract No. 68-02-3890. October 1985.

TABLE VII-4. FIELD CHECKS OF SAMPLING EQUIPMENT

Equipment	Checked against	Allowable difference
Method 5 meter box	Critical orifice	+/-5%
VOST meter box	Bubble meter	+/-5%
Pitot tube	Inspection	No visible damage
Digital indicator	Millivolt signal	+/-0.5%
Thermocouples	ASTM 2F or 3F	+/-1.5%
Balance	Class S weights	+/-0.5 g
Probe nozzles	Caliper	+/-0.004 in.

is purchased for use in the laboratory. Calibration standards are obtained as NBS traceable materials, as primary standards, from the EPA standards repository, or are prepared according to procedures prescribed in the method.

For recurring or common analytical procedures, a known reference standard is routinely analyzed to ensure the continued accuracy of the measurement system. Control charts are prepared using an estimate of the method variability (i.e., standard deviation) obtained from the literature or determined by repeated duplicate analyses run in the laboratory. Corrective action is initiated when the analysis is out of control limits or shows a trend toward bias.

VII.10 Performance and System Audits

The systems audit consists of an on site qualitative inspection and review of the total measurement system. This inspection is expected to be conducted by an EPA observer or contractor assigned to the trial burn test by the EPA or Missouri DNR. During the systems audit, the auditor observes the procedures and techniques of the field team in the following areas:

* setting up and leak testing the sampling train(s)
* isokinetic sampling check of the sampling train
* final leak checks of train(s)
* sample recovery and custody.

Results of the systems audit are summarized in a written report.

Performance audits are conducted during the sampling and analytical phases of the test program. The volumetric flow metering devices are audited during the sampling phase using a critical orifice or bubble flow meter. Barometers, thermometers, nozzles, and other equipment used in sampling are audited to ensure the collection of acceptable data. Appendix E contains audit data sheets to be used.

PEI Associates, Inc. also participates in ongoing performance audit programs conducted by EPA for dry gas test meter calibration (Method 5) and Orsat analysis Method 3).

VII.11 Preventive Maintenance

PEI's preventive maintenance program is composed of three major components consisting of: short interval inspection; replacement of obsolete or damaged components; and scheduled disassembly and overhaul.

The short interval inspection program consists of inspecting each component of the sampling or analytical equipment after each job. This inspection is accomplished after the post-test calibration check or next job calibration, and includes operating and inspecting each component to detect damage.

Replacement of damaged, worn out or obsolete components is made whenever required. For fiel equipment, a red tag system is used which alerts maintenance personnel to damaged or worn out components detected in the field. Spare parts inventories are kept for major field or analytical equipment components. Maintenance logs are kept for each analytical instrument.

Scheduled disassembly and overhaul is done according to manufacturers recommended procedures or to procedures outlined in EPA-APTD-05-76 and EPA-600/4-77-027b.

VII.12 Procedures to Assess Data Precision, Accuracy and Completeness

Acceptable precision is maintained through strict adherence to acceptable limits of difference in replicate measurements at each step of the procedure from initial calibration of sampling equipment to the final analytical determinations. These limits are specified for the method in most instances.

Acceptable accuracy is maintained through rigorous calibration procedures using the standards specified in a method, use of control or reference samples where appropriate, and performance and systems audits.

Acceptable completeness is maintained by following the QA project plan procedures, maintaining a state of control via internal QC checks, and reporting QA/QC results in the final report. All of the above quality indicators will be summarized

and compared to QA objectives and goals in the final report.

VII.13 Corrective Action

The results from the following QA/QC activities may initiate a corrective action:

* performance audits
* system audits
* interlaboratory comparison study
* calibration data out of specified limits
* data completeness below required limits
* failure to adhere to the QA plan
* failure to adhere to SOPs.

The project quality control feedback loop is shown in Figure VII-4. This loop allows for a continuous flow of QA/QC data from each functional unit to the Quality Assurance Coordinator. It is his duty to take early and effective action when data quality falls below required limits.

VII.14 QA Reports to Management

Virtually every project terminates in a written report to the client. Recordkeeping and reporting are key functions of a quality assurance program. Two types of reports used are quality control and quality assessment.

Quality control records are used to document the routine control functions such as calibrations, operation checks, and maintenance activities. Quality assessment records document the precision, accuracy and completeness of the data. Both type of records are compiled by the sample custodian and reviewed by the Quality Assurance Coordinator for completeness and acceptability.

Appendix A

Engineering Drawings

NOZZLE LEGEND

ITEM	QTY.	DESCRIPTION
N1	1	4'-4" OXIDIZER - BURNER PLENUM CONN. - 4'-9" O.D. x ½" R FLG. #760-¾" ø HOLES ON 4'-7" B.C.
N2	1	3'-7" OXIDIZER - BREECHING DUCT - 4'-0" O.D. x ½" R FLG. #752-¾" ø HOLES ON 3'-10" B.C.
N3	1	4" STREAM ø 4 CONN. (4"-150# R.F.S.O.)
N4	1	4" SIGHT PORT/D-ST-0238-3
N5	1	1'-2½" COMBUSTION AIR INLET - 1'-6½" O.D. x ⅜" R FLG. #720-¾" ø HOLES ON 1'-5" B.C.
N6	1	BURNER - BURNER PLENUM CONN. #724 - ⅝" ø BOLTS ON 1'-0½" B.C.
N7	1	4" PILOT CONN. - 7" O.D. x ¼" R FLG. #74 - ⅝" ø HOLES ON 6" B.C.
N8	1	BREECHING DUCT - WATER WEIR CONN. - 4'-5½" O.D. x ½" R FLG #756-¾" ø HOLES ON 4'-3½" B.C.
N9	1	WATER WEIR - CONTACTOR TUBE CONN. - 4'-7" O.D. x ½" R FLG. #756-¾" ø HOLES ON 4'-5" B.C.
N10	1	4'-0" I.D. CONTACTOR TUBE - QUENCH POT CONN. - 4'-7" O.D. FLG. #756 - ¾" ø HOLES ON 4'-5" B.C. (DERAKANE)
N11-N18	8	4" ø QUENCH GUN CONN - (4"-150# F.F.- DERAKANE)
N19	1	2'-2" I.D. QUENCH POT - CONNECTING DUCT CONN. - 2'-9" O.D. FLG. #736 - ¾" ø HOLES ON 2'-7" B.C. (DERAKANE)
N20	1	2" ø INSTRUMENT LEVEL CONN. (3"-150# F.F.- DERAKANE)
N21	1	3" ø INSTRUMENT LEVEL CONN. (2"-150# F.F.- DERAKANE)
N22	1	4" ø EQUALIZER CONN. (4"-150# F.F.- DERAKANE)
N23	1	4" ø DRAIN CONN. (4"-150# F.F.- DERAKANE)
N24	1	2'-2" I.D. CONNECTING DUCT - ABSORBER CONN. - 2'-9" O.D. FLG. #736 - ¾" ø HOLES ON 2'-7" B.C. (DERAKANE)
N25	1	5'-0" I.D. ABSORBER - ABSORBER STACK CONN. - 5'-7" O.D. FLG. #768 - ¾" ø HOLES ON 5'-5" B.C.
N26	1	4" ø ABSORBER EQUALIZER CONN. (4"-150# F.F.- DERAKANE)
N27	1	4" ø DRAIN CONN. (4"-150# F.F.- DERAKANE)
N28	1	4" ø RECYCLE PUMP CONN. (4"-150# F.F.- DERAKANE)
N29	1	2" ø LEVEL CONTROL CONN. (3"-150# F.F.- DERAKANE)
N30	1	3" ø LEVEL CONTROL CONN. (2"-150# F.F.- DERAKANE)
N31	1	3" ø WATER INLET CONN. (3"-150# FF-ERP)
N32	1	6" ø DISTRIBUTOR GUN - ABSORBER CONN. (6"-150# F.F.- DERAKANE)
N33-34	2	4" ø SAMPLE CONN (4"-150# F.F.- DERAKANE)
N35	1	2" SIGHT PORT
N36	1	2" I.D. O₂ ANALYZER (2"-150# F.F.- DERAKANE)
N37,38,39	3	12" I.D. TEMP COUPLE (12"-150# R.F.S.O.
N40-45	6	12" I.D. TEMP COUPLE (12"-150# R.F. DERAKANE)
M1	1	18" MANWAY - 2'-7½" O.D. x ¼" R FLG. #732-¾" ø HOLES ON 2'-5½" B.C.
M2,3,4	3	24" I.D. MANWAY - 2'-1" O.D. FLG. #728-¾" ø HOLES ON 1'-4" B.C.

NOZZLE LEGEND (CONTINUED)

ITEM	QTY.	DESCRIPTION
C1	1	1" PURGE AIR CONN. (1"-N.P.T. FEMALE)
C2,3	2	¾" PURGE AIR CONN. (¾"-N.P.T. FEMALE)
C4-C10	4	1" WEIR WATER CONN. (1"-N.P.T. FEMALE)
C11,C16	2	¾" SCANNER CONN. (¾"-N.P.T. FEMALE)
C17,C18	2	½" PURGE AIR CONN. (½"-N.P.T. FEMALE)
C19	1	2" AUXILIARY GAS CONN. (2"-N.P.T. FEMALE)
C20	1	2" DRAIN CONN #7 CAP (2"-N.P.T. MALE)
C21-C25	3	½" WASTE CONN. (½"-N.P.T. FEMALE)
C26-C29	4	½" STEAM CONN. (½"-N.P.T. FEMALE)
C-30	1	½" FUEL OIL CONN (½"-N.P.T. FEMALE)
C31-C34	4	¾" AIR PURGE CONN. (¾"-N.P.T. FEMALE)
C35,36	2	½" TEMPERING STEAM CONN. (½"-N.P.T. FEMALE)
C37,38	2	½" TEMPERING WATER CONN. (½"-N.P.T. FEMALE)
C39,40	2	2" TEMPERING GUN CONN. 2"-N.P.T. MALE)
C41	1	1" AIR PURGE CONN. (1"-N.P.T. FEMALE)
C42	1	½" PILOT GAS CONN. (½"-N.P.T. FEMALE)
C43	1	½" CONDUIT CONN. (½"-N.P.T. FEMALE)

GENERAL NOTES

1. ALL PLATE TO BE A-283 GR. 'C' OR BETTER UNLESS OTHERWISE NOTED
2. ALL BOLT HOLES TO STRADDLE NORMAL C's. UNLESS OTHERWISE NOTED
3. ALL GLASS FIBER MATERIALS OF CONSTRUCTION TO BE FEP TEFLON LINED (90 MILS MIN THK.) GLASS FIBER REINFORCED DERAKANE 470-45 VINYL ESTER RESIN CONSTRUCTION UNLESS OTHERWISE NOTED.
4. ALL REFRACTORY TO BE COMBUSTION ENGINEERING REFRACTORIES CO. OR EQUAL UNLESS OTHERWISE NOTED.
5. ALL REFRACTORY TO BE SUPPLIED AND SHOP INSTALLED PER JZ CO. SPEC. # 100, 110, 120
6. ALL GASKET MATERIAL TO BE BUNA 'N' UNLESS NOTED OTHERWISE
7. ALL MK. NO's. TO BE PRECEDED BY S.O. NO.
8. FINISH EXTERIOR: ALL CARBON STEEL SURFACES TO BE SANDBLASTED IN ACCORDANCE W/ SPEC. # SSPC-SP-10-63T AND PAINTED W/ PRIMER COAT OF CARBOLINE CO's CARBO-ZINC #11 (GREEN) (2.5-3.5 M.S. DFT.) AND ONE SEAL COAT OF CARBOLINE CO's CARBOLINE # 4674 (ALUM. AND BLACK) (1-1.5 MILS D.F.T.) UNLESS OTHERWISE NOTED. MINIMUM ACCEPTABLE TOTAL THICKNESS IS 4 MILS D.F.T. SILICONE SEAL COAT TO BE SUPPLIED BY JOHN ZINK CO., AND APPLIED BY OTHERS IN FIELD.

REFERENCE DRAWINGS

- B-O-81181-202 : PLAN VIEW - GENERAL ASSY.
- B-O-81181-203 : SIDE ELEV. - GENERAL ASSY.
- B-O-81181-204 : END ELEV. - GENERAL ASSY.
- B-O-81181-205 : ANCHOR BOLT PLAN
- B-O-81181-301 : QUENCH POT DETAIL
- B-O-81181-302 : CONTACTOR TUBE DETAIL
- B-O-81181-303 : WATER WEIR DETAIL
- B-O-81181-304 : BREECHING DUCT DETAIL
- B-O-81181-305 : BURNER PLENUM DETAIL
- B-O-81181-306 : THERMAL OXIDIZER DETAIL
- B-O-81181-307 : ABSORBER (LOWER SECTION)
- B-O-81181-308 : ABSORBER (UPPER SECTION)
- B-O-81181-309 : CONNECTING DUCT
- B-O-81181-310 : PLATFORM DETAILS

- B-O-81181-601 : HI-24 BURNER
- B-O-81181-602 : TEMPERING GUN
- A-O-81181-603 : EA WASTE GUN INSERT
- A-O-81181-604 : DH WASTE GUN INSERT
- A-O-81181-605 : EA OIL GUN INSERT
- B-ST-0065-2 : B-ST-0065 PILOT ASS'Y
- D-ST-0238-3 : SIGHT PORT ASS'Y
- CA-O-81181-801 : CAPACITY CURVE
- CA-ST-0027-2 : RZ TYPE OIL BODY ASS'Y
- CA-ST-0028-3 : RZ TYPE OIL BODY ASS'Y
- B-ST-0062-3 : PLATFORM STD. DETS.
- B-ST-0064-3 : LADDER STD. DETS.
- A-ST-0001-2 : MIST PAD INST. DETS.
- B-O-81181-606 : CONTACTOR GUN ASS'Y
- A-O-81181-607 : DH WASTE GUN INSERT

MONSANTO COMPANY — Order No. 3008-9-0037
CORPORATE ENGR. DEPT. Project (CEA) No. 3008
Location ST. Louis, Mo. Area 09
Equipment No. 111.004, 114.006, 165.060
Description Oxidizer System
Reviewed by [illegible] Date 4-23-76
☐ Mate Confirmation Required ☐ Leave on Sheet ☒

NO.	REVISION OR SCRIPTION	UNIT BY	CK	APP	DATE
1	REVISED PER CUST. REQUEST & ECN #1266	JW	dh	CP	2-13-76

MONSANTO — ST. LOUIS, MO. — C81181 — 3008-9-0037

JOHN ZINK COMPANY
TULSA OKLAHOMA

NOZZLE LEGEND, GENERAL NOTES AND REFERENCE DWGS.

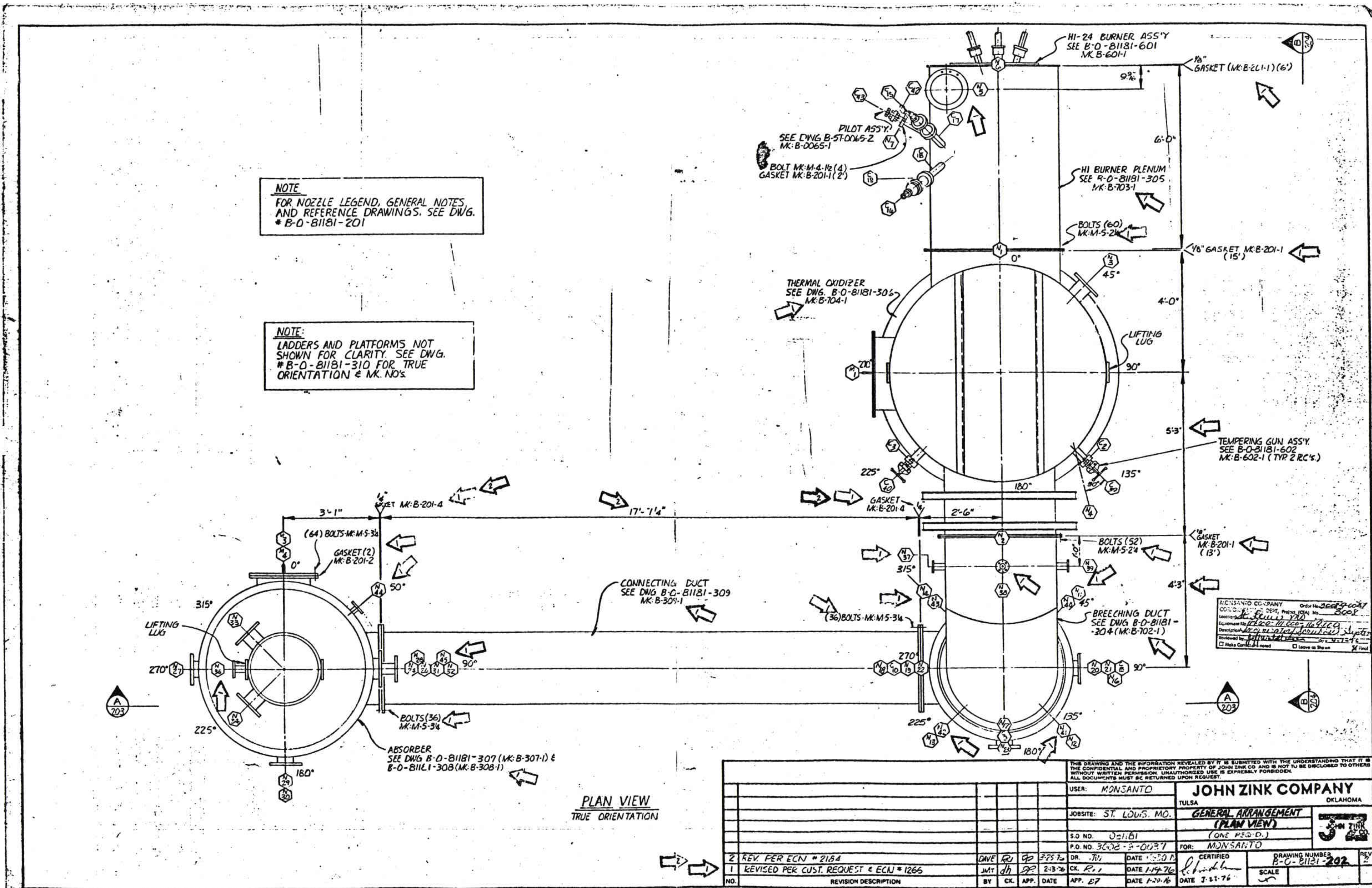
FOR MONSANTO

DRAWN JW 1-6-76
ENGR. PJ 1-19-76
APP. [illegible]

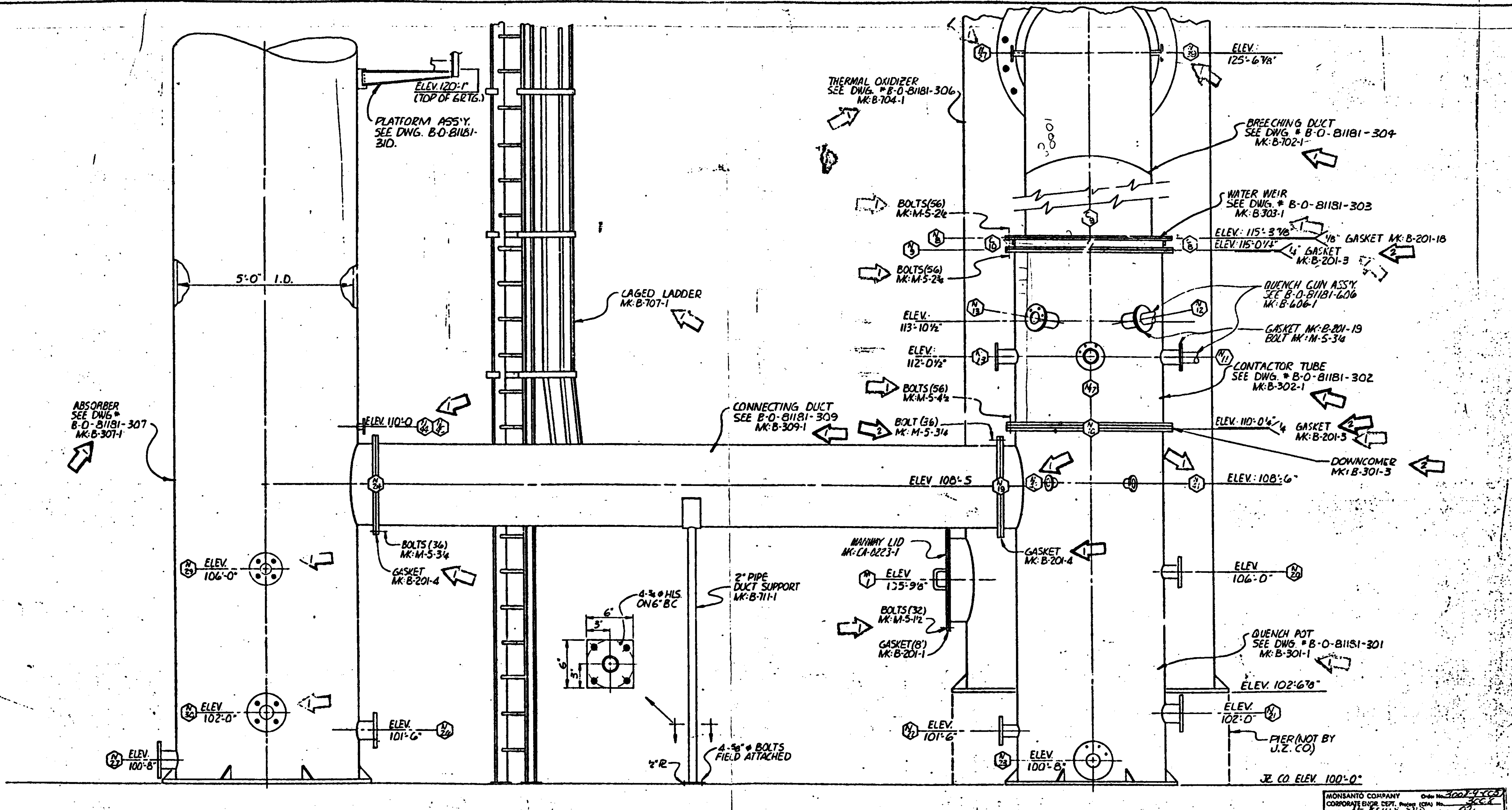
CERTIFIED [signature] DATE 3-22-76

DRAWING NUMBER B-O-81181-201 REV 1

SCALE



HI-24 BURNER ASS'Y
SEE B-0-81181-601
MK B-601-1
1/8" GASKET (MK B-201-1) (6')
9 3/4"
6'-0"
PILOT ASS'Y.
SEE DWG B-57-0065-2
MK: B-0065-1
BOLT MK:M-4-1½ (4)
GASKET MK: B-201-1 (2')
HI BURNER PLENUM
SEE B-0-81181-305
MK B-703-1
BOLTS (60)
MK:M-5-2¼
1/8" GASKET MK:B-201-1
(15')
NOTE
FOR NOZZLE LEGEND, GENERAL NOTES, AND REFERENCE DRAWINGS, SEE DWG. # B-0-81181-201
NOTE:
LADDERS AND PLATFORMS NOT SHOWN FOR CLARITY. SEE DWG. # B-0-81181-310 FOR TRUE ORIENTATION & MK. NO's.
THERMAL OXIDIZER
SEE DWG. B-0-81181-306
MK:B-704-1
4'-0"
LIFTING LUG
5'-3"
TEMPERING GUN ASS'Y.
SEE B-0-81181-602
MK:B-602-1 (TYP. 2 RC's.)
GASKET MK:B-201-4
3'-1"
17'-7¼"
2'-6"
(64) BOLTS-MK:M-5-3¼
GASKET (2)
MK:B-201-2
BOLTS (52)
MK:M-5-2¼
1/8" GASKET
MK:B-201-1
(13')
4'-3"
CONNECTING DUCT
SEE DWG B-0-81181-309
MK:B-309-1
(36) BOLTS-MK:M-5-3¼
BREECHING DUCT
SEE DWG B-0-81181-304 (MK:B-702-1)
LIFTING LUG
BOLTS (36)
MK:M-5-3¼
ABSORBER
SEE DWG B-0-81181-307 (MK:B-307-1) & B-0-81181-308 (MK:B-308-1)
PLAN VIEW
TRUE ORIENTATION
MONSANTO COMPANY
THIS DRAWING AND THE INFORMATION REVEALED BY IT IS SUBMITTED WITH THE UNDERSTANDING THAT IT IS THE CONFIDENTIAL AND PROPRIETARY PROPERTY OF JOHN ZINK CO AND IS NOT TO BE DISCLOSED TO OTHERS WITHOUT WRITTEN PERMISSION. UNAUTHORIZED USE IS EXPRESSLY FORBIDDEN. ALL DOCUMENTS MUST BE RETURNED UPON REQUEST.
USER: MONSANTO
JOBSITE: ST. LOUIS, MO.
S.O. NO. 0-81181
P.O. NO. 3608-5-0037
JOHN ZINK COMPANY
TULSA OKLAHOMA
GENERAL ARRANGEMENT
(PLAN VIEW)
(ONE REQ'D.)
FOR: MONSANTO
DRAWING NUMBER B-0-81181-202
REV 2
2 REV. PER ECN # 2164 DAVE 3-25-76
1 REVISED PER CUST. REQUEST & ECN # 1266 2-13-76
NO. REVISION DESCRIPTION BY CK. APP. DATE
DATE 1-14-76
DATE 1-20-76
DATE 3-23-76
CERTIFIED
SCALE



SIDE ELEVATION (A/202)

NOTE
FOR NOZZLE LEGEND, GENERAL NOTES, AND REFERENCE DRAWINGS SEE DWG. # B-0-81181-201

NO.	REVISION DESCRIPTION	BY	CK.	APP.	DATE
2	REV. PER ECN # 2184	DAVE	[illegible]	[illegible]	5-25-76
1	REVISED PER CUSTOMER REQUEST ECN # 1266	WRB	[illegible]	[illegible]	2-13-76

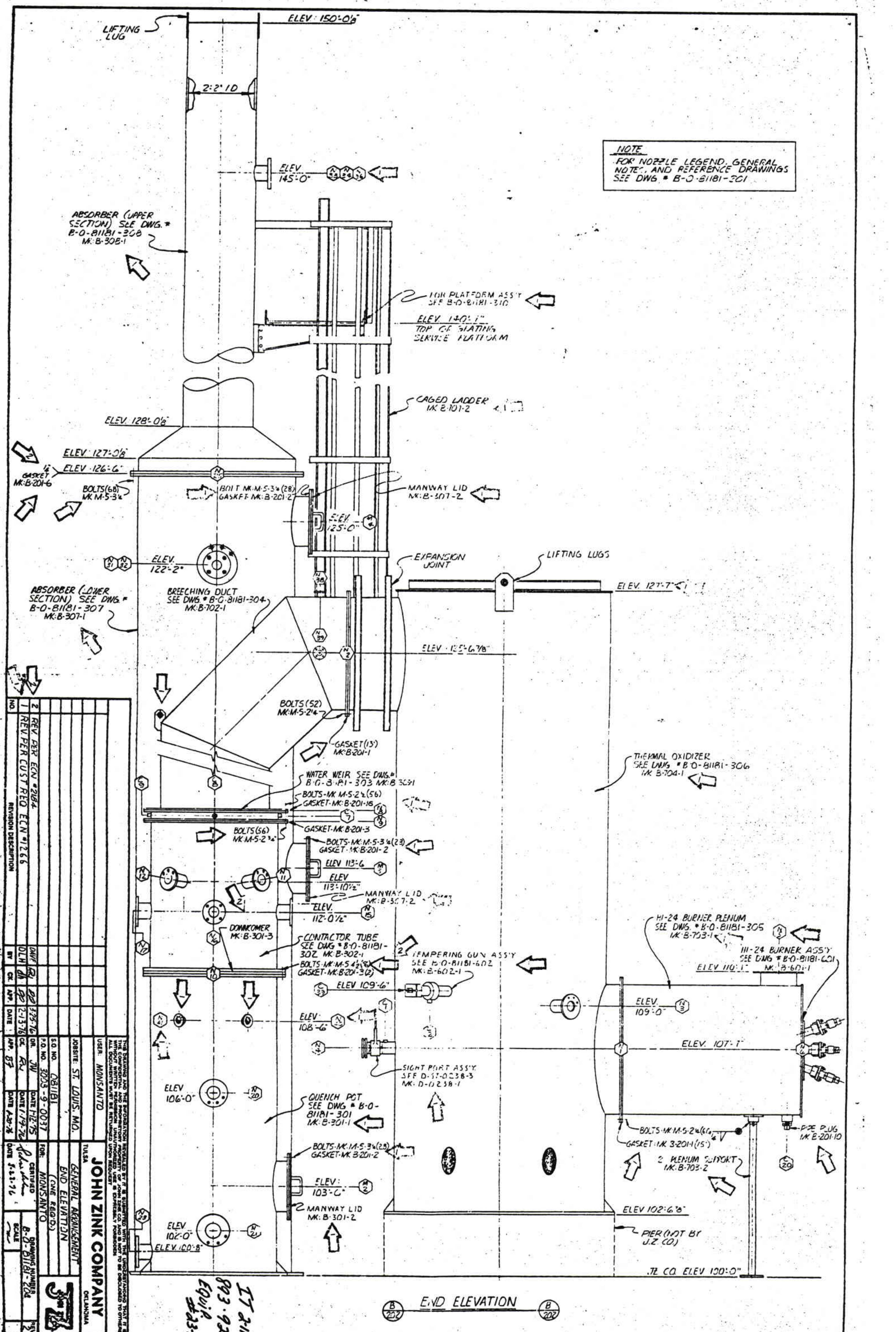
USER: MONSANTO
JOBSITE: ST. LOUIS, MO.
S.O. NO. 381161
P.O. NO. 3008-9-0057

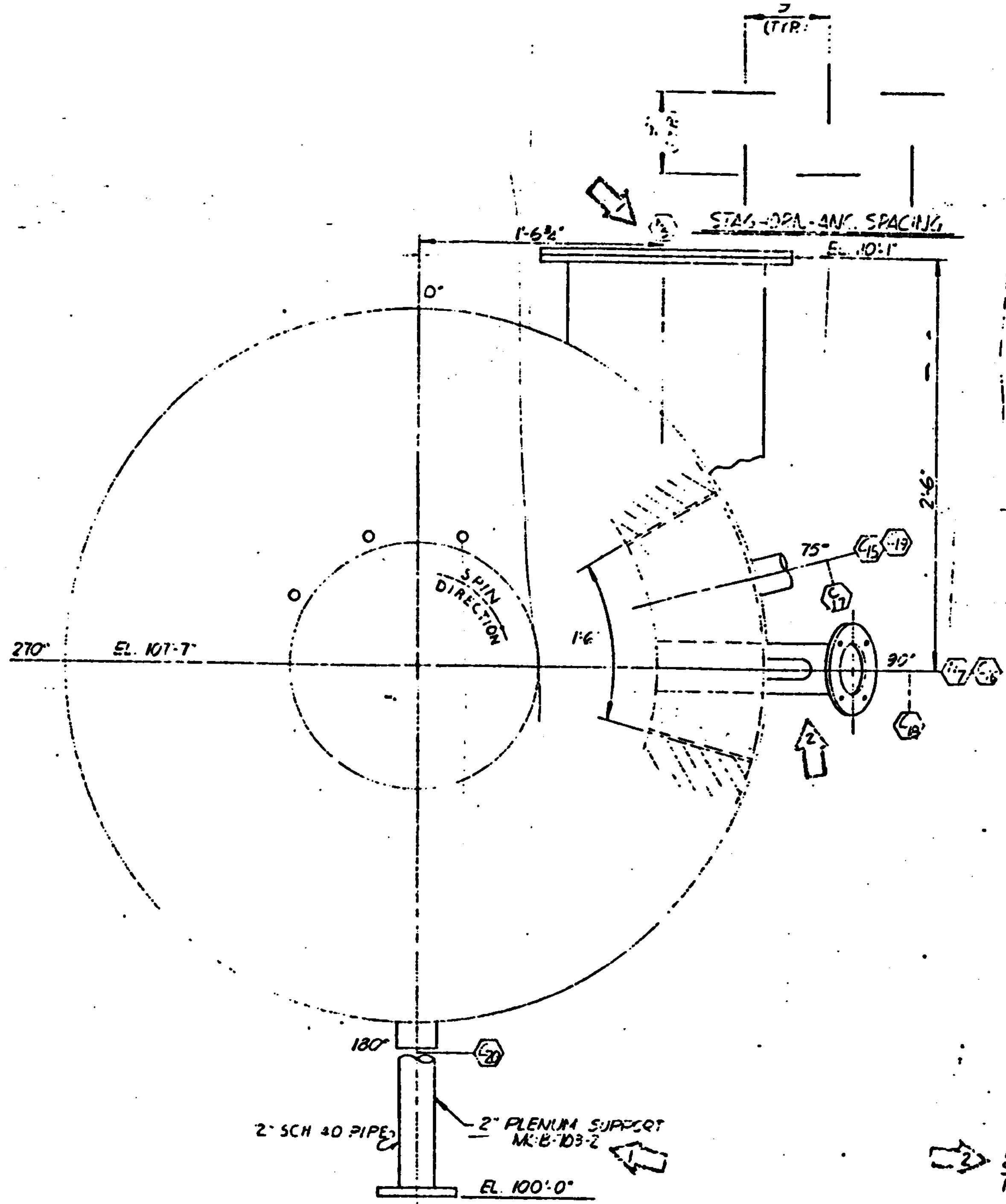
JOHN ZINK COMPANY
TULSA OKLAHOMA

GENERAL ARRANGEMENT
SIDE ELEVATION
(ONE REQ'D.)
FOR: MONSANTO

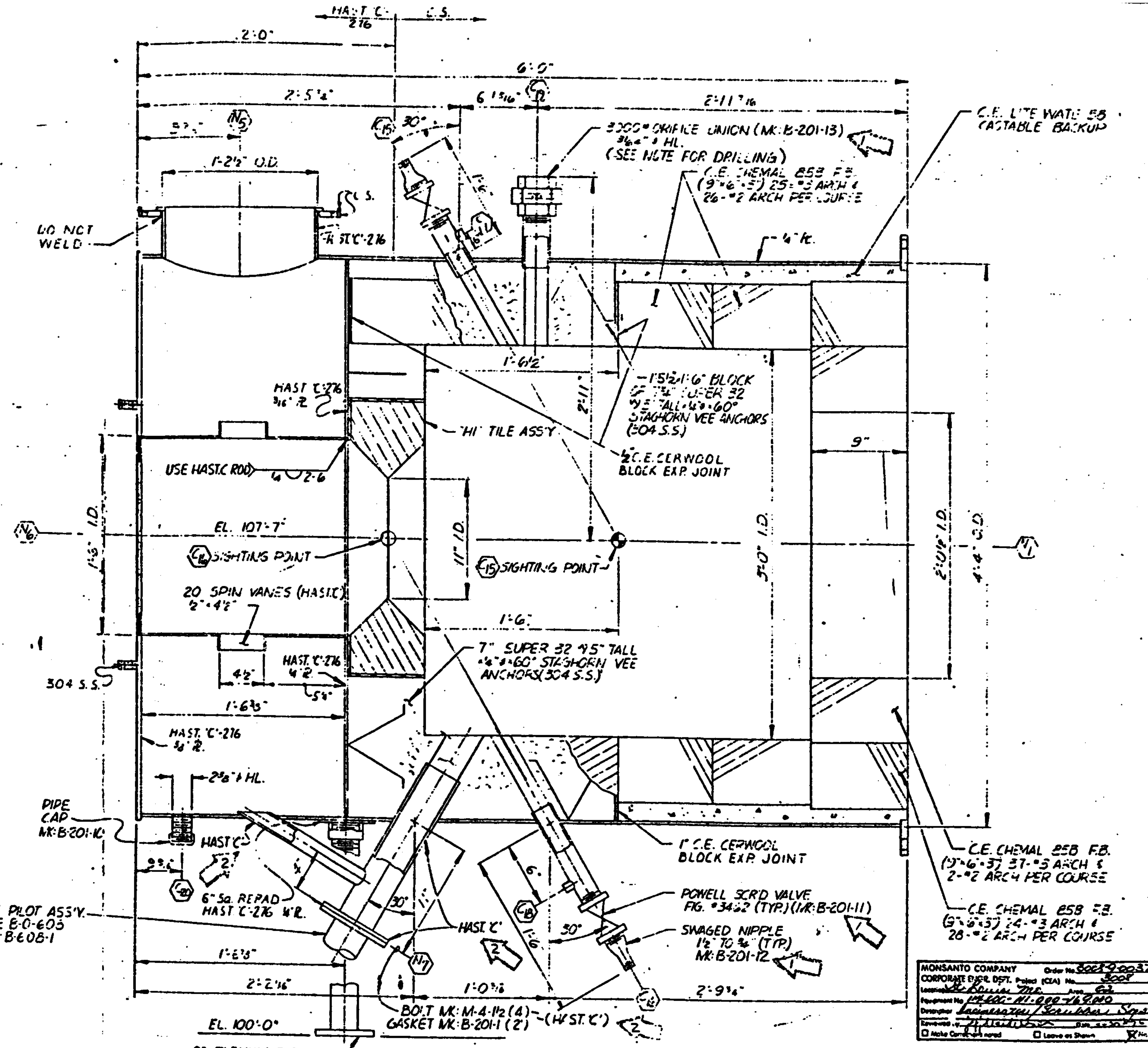
DR. TN	DATE 1-10-76
CK. R.J	DATE 1-19-76
APP. E.F	DATE 1-29-76

CERTIFIED [signature] DATE 3-23-76
DRAWING NUMBER B-0-81181-203 REV 2
SCALE


LIFTING LUG
ELEV: 150'-0⅛"
2'-2" I.D.
ELEV 145'-0"
ABSORBER (UPPER SECTION) SEE DWG. # B-0-81181-308 MK: B-308-1
FOR PLATFORM ASS'Y SEE B-0-81181-310
ELEV. 140'-1" TOP OF GRATING SERVICE PLATFORM
NOTE
FOR NOZZLE LEGEND, GENERAL NOTES, AND REFERENCE DRAWINGS SEE DWG # B-0-81181-301
CAGED LADDER MK B-101-2
ELEV. 128'-0⅛"
ELEV. 127'-0⅛"
ELEV. 126'-6"
¼" GASKET MK: B-201-6
BOLTS(68) MK M-5-3¾
BOLT MK: M-5-3¼ (28)
GASKET MK: B-201-2
MANWAY LID MK: B-307-2
ELEV. 125'-0"
ELEV. 122'-2"
EXPANSION JOINT
LIFTING LUGS
ELEV. 127'-7"
ABSORBER (LOWER SECTION) SEE DWG # B-0-81181-307 MK: B-307-1
BREECHING DUCT SEE DWG # B-0-81181-304 MK: B-702-1
ELEV. 125'-6⅞"
BOLTS (52) MK: M-5-2¼
GASKET (13') MK: B-201-1
THERMAL OXIDIZER SEE DWG # B-0-81181-306 MK B-704-1
WATER WEIR SEE DWG # B-0-81181-303 MK: B-303-1
BOLTS-MK M-5-2½ (56)
GASKET-MK: B-201-18
GASKET-MK: B-201-3
BOLTS (56) MK: M-5-2¾
BOLTS-MK: M-5-3¼ (28)
GASKET-MK: B-201-2
ELEV 113'-6
ELEV 113'-10½"
MANWAY LID MK: B-307-2
ELEV. 112'-0½"
DOWNCOMER MK: B-301-3
CONTACTOR TUBE SEE DWG # B-0-81181-302 MK: B-302-1
BOLTS-MK: M-5-4½ (8)
GASKET-MK: B-201-3 (2)
TEMPERING GUN ASS'Y SEE B-0-81181-602 MK: B-602-1
HI-24 BURNER PLENUM SEE DWG. # B-0-81181-305 MK: B-703-1
HI-24 BURNER ASS'Y SEE DWG # B-0-81181-601 MK: B-601-1
ELEV 110'-1"
ELEV 109'-6"
ELEV. 109'-0"
ELEV 108'-6
ELEV. 107'-1"
SIGHT PORT ASS'Y SEE D-ST-0238-3 MK: D-0238-1
ELEV 106'-0"
QUENCH POT SEE DWG # B-0-81181-301 MK: B-301-1
BOLTS-MK: M-5-3¼ (28)
GASKET-MK B-201-2
BOLTS-MK M-5-2¼ (60)
GASKET MK B-201-1 (15')
PIPE PLUG MK B-201-10
2 PLENUM SUPPORT MK: B-703-2
ELEV: 103'-0"
MANWAY LID MK: B-301-2
ELEV 102'-6⅜"
PIER (NOT BY J.Z. CO.)
ELEV 102'-0"
ELEV. 100'-8"
J.Z. CO. ELEV 100'-0"
END ELEVATION
B 202
NO. | REVISION DESCRIPTION | BY | CK. | APP. | DATE
2 | REV. PER ECN #2184 | DAVE | RJ | DD | 3-25-76
1 | REV. PER CUST REQ ECN #1266 | DLH | dh | DD | 2-13-76
THIS DRAWING AND THE INFORMATION REVEALED BY IT IS SUBMITTED WITH THE UNDERSTANDING THAT IT IS THE CONFIDENTIAL AND PROPRIETARY PROPERTY OF JOHN ZINK CO. AND IS NOT TO BE DISCLOSED TO OTHERS WITHOUT WRITTEN PERMISSION. UNAUTHORIZED USE IS EXPRESSLY FORBIDDEN. ALL DOCUMENTS MUST BE RETURNED UPON REQUEST.
USER: MONSANTO
JOBSITE: ST. LOUIS, MO.
S.O. NO. 081181
P.O. NO. 3003-9-0037
DR. JW DATE 11-12-75
CK. RJ DATE 1-19-76
APP. BB DATE 1-20-76
JOHN ZINK COMPANY
TULSA OKLAHOMA
GENERAL ARRANGEMENT
END ELEVATION
(ONE REQ'D.)
FOR: MONSANTO
CERTIFIED
DATE 5-23-76
SCALE
DRAWING NUMBER B-0-81181-204
REV 2
IT 2114
893.92
Equip. #23-2059



END VIEW
(TRUE ORIENTATION)



PART SECTIONAL ELEVATION

STL. WT: 14 KIPS
REFR. WT: 70 KIPS
TOTAL WT: 84 KIPS
MK: B-703-1-ONE REQ'D.

NOTE: FOR NOZZLE LEGEND, GENERAL NOTES, & REFERENCE DWG'S., SEE 5-D-3181-201

NOTE:
AUXILIARY FUEL GAS HEAT RELEASE
5,000,000 BTU/HR @ 10 PSIG (PRELIMINARY)
HEATING VALUE: 9?5 BTU/SCF
SPECIFIC GRAVITY: .55
DRILLING: DRILL ONE #/64 PORT IN ORIFICE PLATE

REFRACTORY QUANTITIES

QTY.	DESCRIPTION	% WEE
191	CHEMAL 85B F.B. 9"×6"×3" #3 ARCH	2%
164	CHEMAL 85B F.B. 9"×6"×3" #2 ARCH	2%
5 FT.	CERWOOL BLOCK 1" THK × 12"×30"	5%
332#	SUPER CASTABLE #32	10%
517#	LITE WATE 55 CASTABLE	25%
255	CHEMAL 111 MORTAR	5%

NO	REVISION DESCRIPTION	BY	CK.	APP	DATE
2	REVISED PER ECN #2200				4-3-76
1	REVISED PER CUST. REQUEST & ECN #2?6				

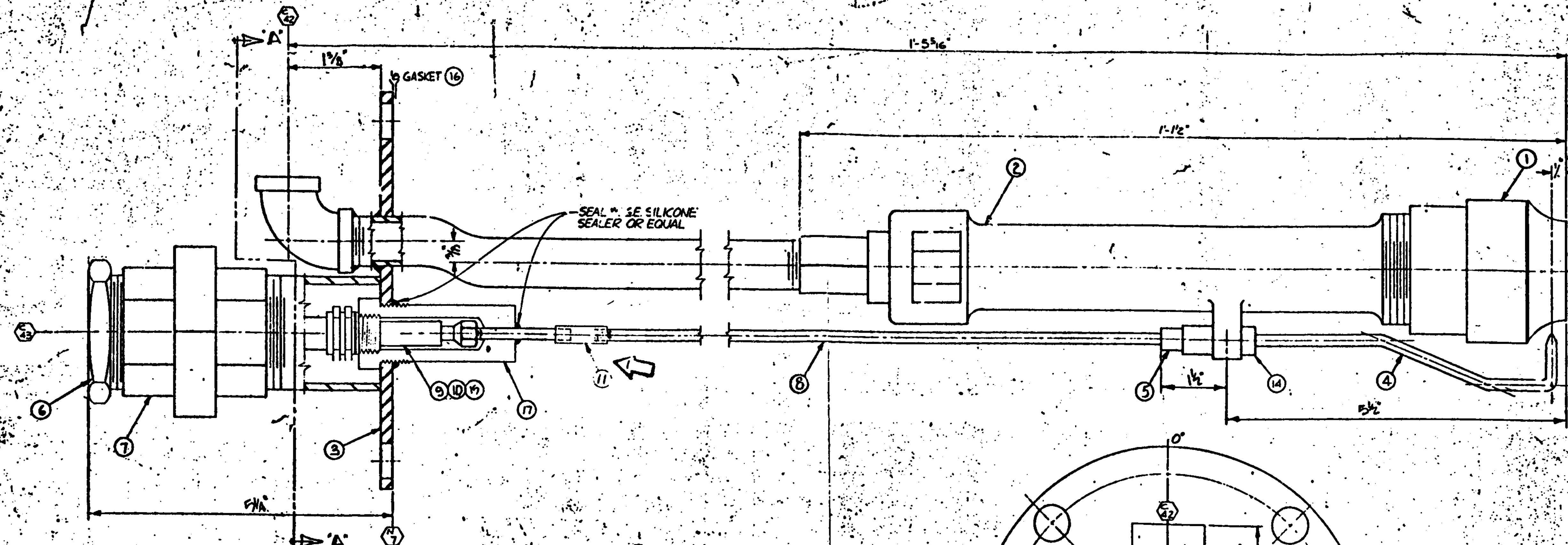
USER MONSANTO
JOBSITE
SO NO
PO NO.
DR / DATE
CK. / DATE
APP. / DATE

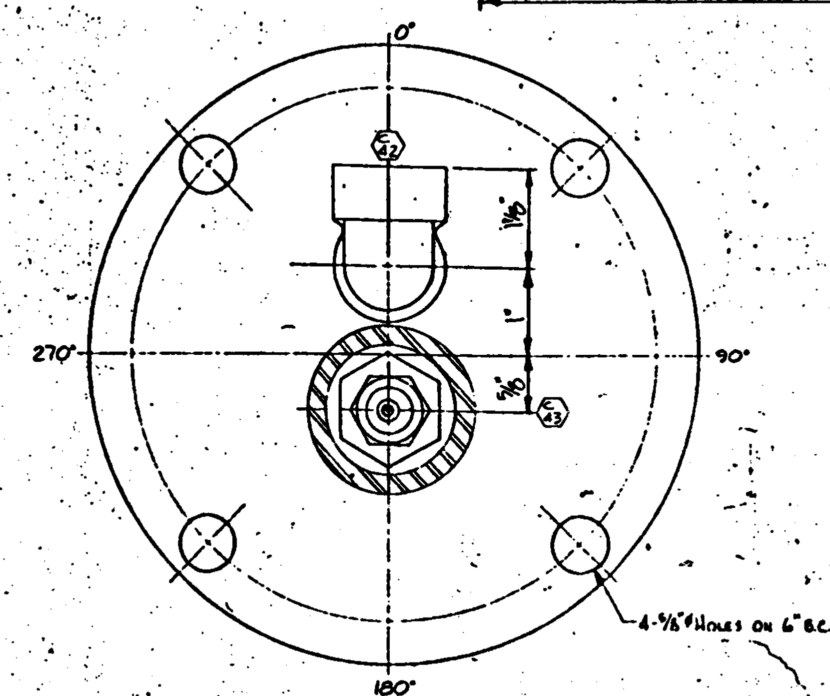
JOHN ZINK COMPANY
TULSA OKLAHOMA

HI-24 BURNER PLENUM
REFRACTORY DETAIL

FOR:
CERTIFIED
DATE
DRAWING NUMBER 305
REV 2
SCALE

MONSANTO COMPANY
CORPORATE ENGR. DEPT.





SECTION 'A-A'

Parts List Per Assembly				
Item	Qty	Description	Part N°	Mat'L
1	1	Pilot Tip (ST-6-1½)	CA-ST-0026	HAST 'C'
2	1	Pilot Mixer Assembly (ST-6-1½-AHR)	CA-ST-0282	HAST 'C'
3	1	Mounting Flange Ass'y	A-0-713	HAST 'C'
4	1	Ignition Rod	CA-ST-D100	HAST 'C'
5	1	Connecting Coupling	[illegible]	HAST 'C'
6	1	Pipe Bushing (Hex Head)	½" × 1½" 3000	STEEL
7	1	Pipe Union (SCREWED)	1½" 3000	STEEL
8	1	Ignition Rod Extension	5/16" ∅	HAST 'C'
9	1	Spark Plug	12-WCC-12-1K	—
10	1	Snap On Connector	[illegible]	—
11	1	[illegible]	[illegible]	[illegible]
12				
13				
14	1	Insulator	32E-27782	CERAMIC
15	1	Terminal Nut	CHAMP #A 543	—
16	1	Gasket	⅛" Thk.	ASB.
17	1	CORROSION SEAL	A-0-713	TEFLON

NOZZLE LEGEND		
ITEM	QUAN	DESCRIPTION
N7	1	4" PILOT CONN. W/ 4-⅝" ∅ H/S ON 6" B.C.
C42	1	½" PILOT GAS CONN. - ½" N.P.T. FEMALE
C43	1	½" CONDUIT CONN. - ½" N.P.T. FEMALE

MONSANTO COMPANY Order No. 3 7B4-0037
CORPORATE ENGR. DEPT. Project (CIA) No. 3029
Location [illegible] Area C2
Equipment No. 02-114.000
Description BURNER PILOT ASSEMBLY
Reviewed by [illegible] Date 6-1?-76
☐ Make Corrections Noted ☐ Leave as Shown ☒ Final

NO	REVISION DESCRIPTION	BY	CK.	APP.	DATE
1	[illegible] ITEM #11 (AS BUILT)			DP	

USER: MONSANTO

JOBSITE: ST. LOUIS, MO.

S.O NO. 081181

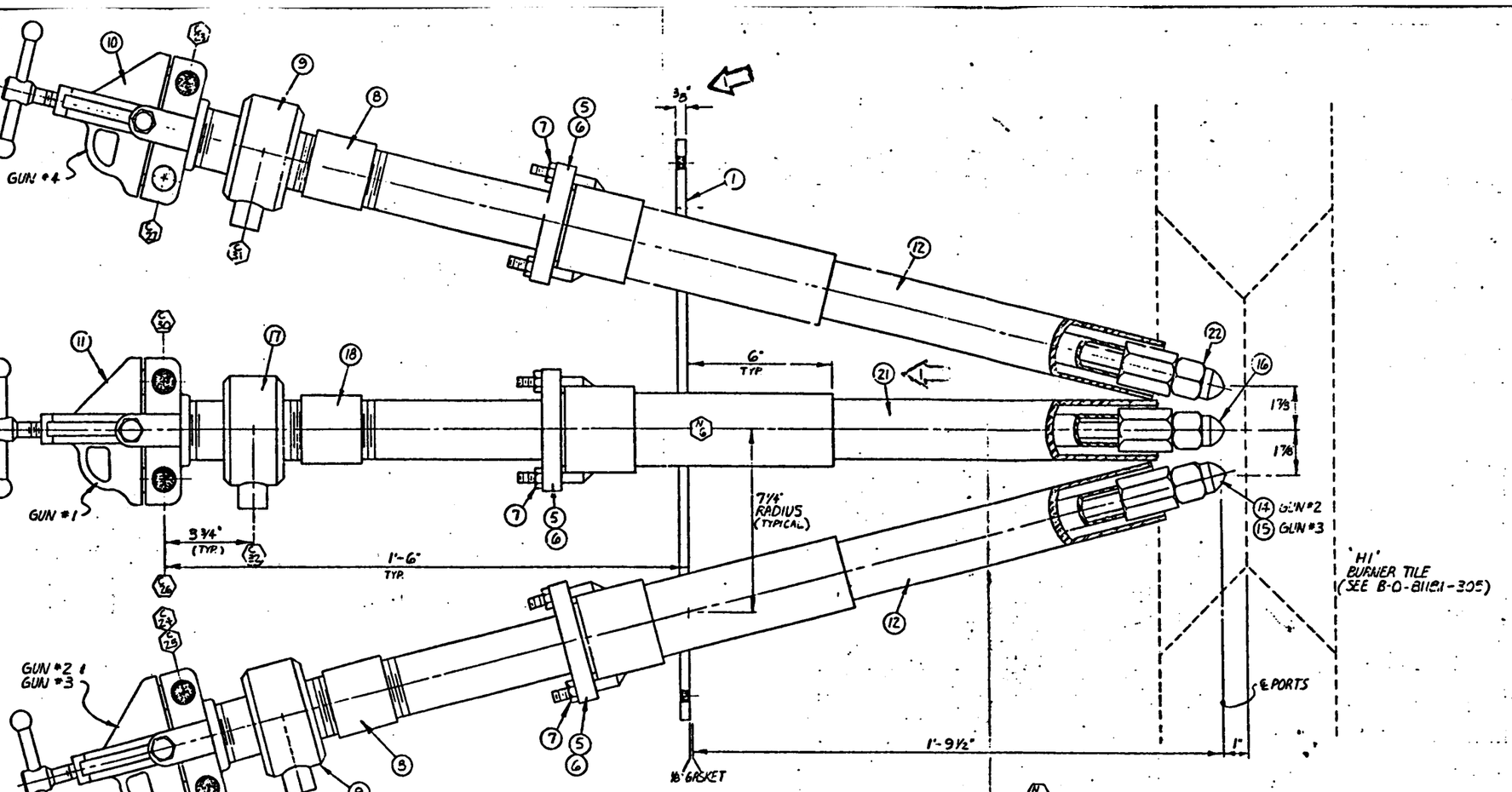
P.O NO. 3008-9-0037

JOHN ZINK COMPANY
TULSA OKLAHOMA

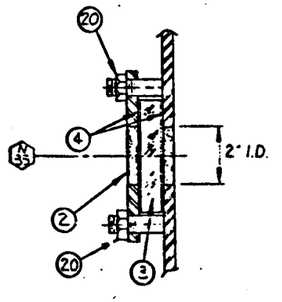
JOHN ZINK "HT" PILOT ASSEMBLY W/ 3" GUIDE TUBE

FOR: MONSANTO

DR. [illegible]	DATE 4-?-76	CERTIFIED	DRAWING NUMBER	REV
CK. [illegible]	DATE [illegible]	[illegible]	B-0-81181-608	1
APP. DP	DATE 4-13-76	DATE ?/31/76	SCALE [illegible]	



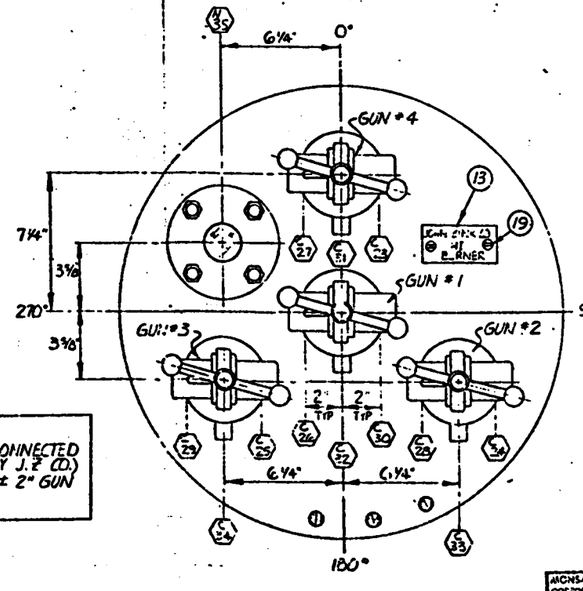
PART SECTIONAL ELEVATION
ONE-MK B-601-1



SIGHT PORT DETAIL

NOTE:
ON GUN #1 AIR SEAL A CONTINUOUS PURGE IS REQ'D. @ 25 P.S.I.G. MIN.

NOTE:
ALL CONNECTIONS TO BE CONNECTED W/ FLEX HOSE. (NOT BY J.Z. CO.) PIPING TO ALLOW FOR ± 2" GUN MOVEMENT



END VIEW
TRUE ORIENTATION

PARTS LIST (PER BURNER)

ITEM	QUAN.	DESCRIPTION	PART NO	MATL.
1	1	FRONT PLATE ASS'Y.	B-0-208	HAST'C
2	1	SIGHT PORT COVER	—	A-285C
3	1	SIGHT GLASS (4" O.D. x ¾" THK.)	—	PYREX
4	2	GASKET (4" O.D. x 2½" I.D. x 1/16" THK.)	—	BUNA'N
5	4	PACKING GLAND	B-108-2	HAST.C
6	4 FT	GARLOCK ROPE - ½"ø	—	ASB.
7	8	⅝" HEX NUTS	—	A-307
8	3	2" STD. WT COUPLING	2"	HAST.C
9	3	AIR SEAL ASS'Y. / D-ST-0014		HAST.C
10	3	WASTE BODY ASS'Y. / CA-ST-0028-3	P2-56	HAST.C
11	1	WASTE BODY ASS'Y. / CA-ST-0027-2	A2-56	DUCT.
12	3	2" GUIDE TUBE (2⅜" O.D. x 1⅞" I.D.)	—	HAST.C
13	1	JOHN ZINK NAMEPLATE	—	ALUM.
14	1	EA GUN INSERT / A-0-81181-603	—	—
15	1	DH GUN INSERT / A-0-81181-604	—	—
16	1	EA GUN INSERT / A-0-81181-605	—	—
17	1	AIR SEAL ASS'Y	D-ST-0014	STL.
18	1	2" STD. WT. COUPLING	2"	A-197
19	2	#9 DRIVE SCREWS	#9 x ⅜"	—
20	4	⅝" HEX NUTS	—	A-307
21	1	2" GUIDE TUBE (2⅜" O.D. x 1⅞" I.D.)	—	HAST.C
22	1	DH GUN INSERT / A-0-81181-607	—	—

NOZZLE LEGEND

ITEM	QUAN.	DESCRIPTION
N6	1	BURNER-BURNER PLENUM CONN. W/ 24 - ⅝"ø HOLES ON 1'-0" B.C.
N35	1	2" SIGHT PORT
C-26-29	4	½" STEAM CONN. (½"-N.P.T. FEMALE)
C-30	1	½" FUEL OIL CONN. (½"-N.P.T. FEMALE)
C-23-25	3	½" WASTE CONN. (½"-N.P.T. FEMALE)
C-31-34	4	⅜" AIR PURGE CONN. (⅜"-N.P.T. FEMALE)

HEAT RELEASE AND FLOW RATES

GUN #1 ~ (#2 FUEL OIL) 5.5 M BTU/HR MAX @ 70 PSIG, 100 PSIG STEAM. 1.1 M BTU/HR MIN. @ 30 PSIG, 60 PSIG STEAM.

GUN #2 ~ 45 PSIG LIQUID PRESSURE, 80 PSIG STEAM PRESSURE MONSANTO STREAM #1

GUN #3 ~ 60 PSIG LIQUID PRESSURE, 90 PSIG STEAM PRESSURE MONSANTO STREAM #2

GUN #4 ~ 60 PSIG LIQUID PRESSURE, 90 PSIG STEAM PRESSURE MONSANTO STREAM #3

GENERAL NOTES

1. ALL BOLT HOLES TO STRADDLE NORMAL C.L. UNLESS OTHERWISE NOTED.
2. FINISH EXTERIOR: ALL C.S. SURFACES TO BE PAINTED W/ ONE COAT OF HIGH TEMP. ALUM.
3. ALL MK. NO'S TO BE PRECEDED W/ S.O. NO.

REFERENCE DRAWINGS

B-0-81181-305: HI BURNER PLENUM
A-0-81181-603: 'EA' GUN INSERT (GUN #2)
A-0-81181-604: 'DH' GUN INSERT (GUN #3)
A-0-81181-605: 'EA' GUN INSERT (GUN #1)
A-0-81181-607: 'DH' GUN INSERT (GUN #4)

REV. NO.	
4	
3	
2	
1	REVISED AS NOTED PER ECN #1266, #1275

THIS DRAWING AND THE INFORMATION REVEALED BY IT IS SUBMITTED WITH THE UNDERSTANDING THAT IT IS THE CONFIDENTIAL AND PROPRIETORY PROPERTY OF JOHN ZINK CO. AND IS NOT TO BE DISCLOSED TO OTHERS WITHOUT WRITTEN PERMISSION. UNAUTHORIZED USE IS EXPRESSLY FORBIDDEN. ALL DOCUMENTS MUST BE RETURNED UPON REQUEST

USER: MONSANTO

JOHN ZINK COMPANY — TULSA, OKLAHOMA

JOBSITE: ST. LOUIS, MO.

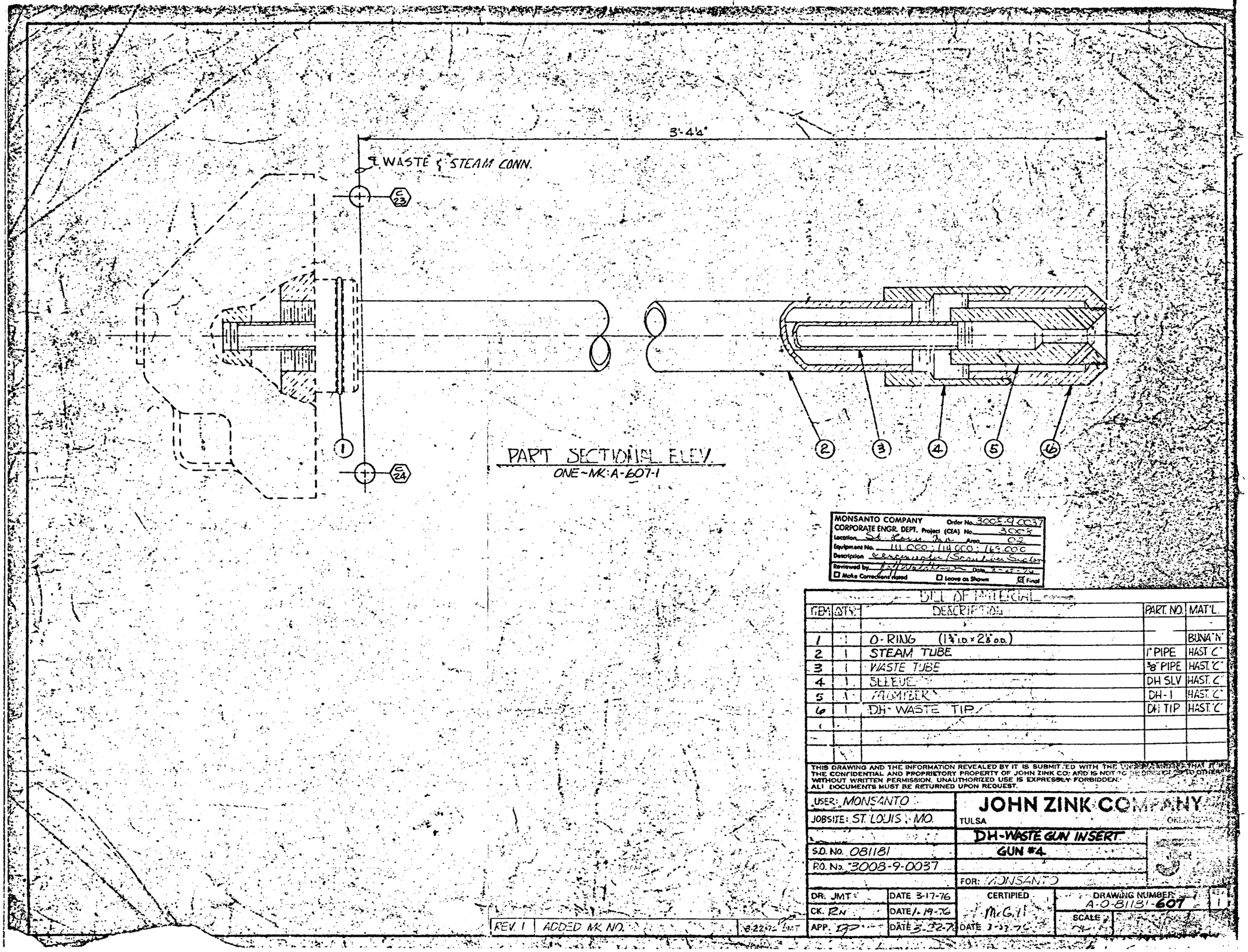
HI-24 BURNER ASSY.

S.O. NO. 08181

P.O. NO. 3009-9-00537 FOR: MONSANTO

DR. JW	DATE 1-2-76	CERTIFIED	DRAWING NUMBER B-0-81181-601 REV 1
CK. RW	DATE 1-19-76		
APP.	DATE 1-20-76	DATE 2-23-76	SCALE

MONSANTO COMPANY
CORPORATE ENGR. DEPT.



PART SECTIONAL ELEV.
ONE-MK:A-607-1

MONSANTO COMPANY Order No. 3008-9-0037
CORPORATE ENGR. DEPT. Project (CEA) No. 3008
Location St Louis, Mo Area 02
Equipment No. 111 000; 114 000; 145 000
Description Decoupler/Scrubber System
Reviewed by J. Fowler Date 2-25-76
☐ Make Corrections Noted ☐ Leave as Shown ☑ Final

BILL OF MATERIAL

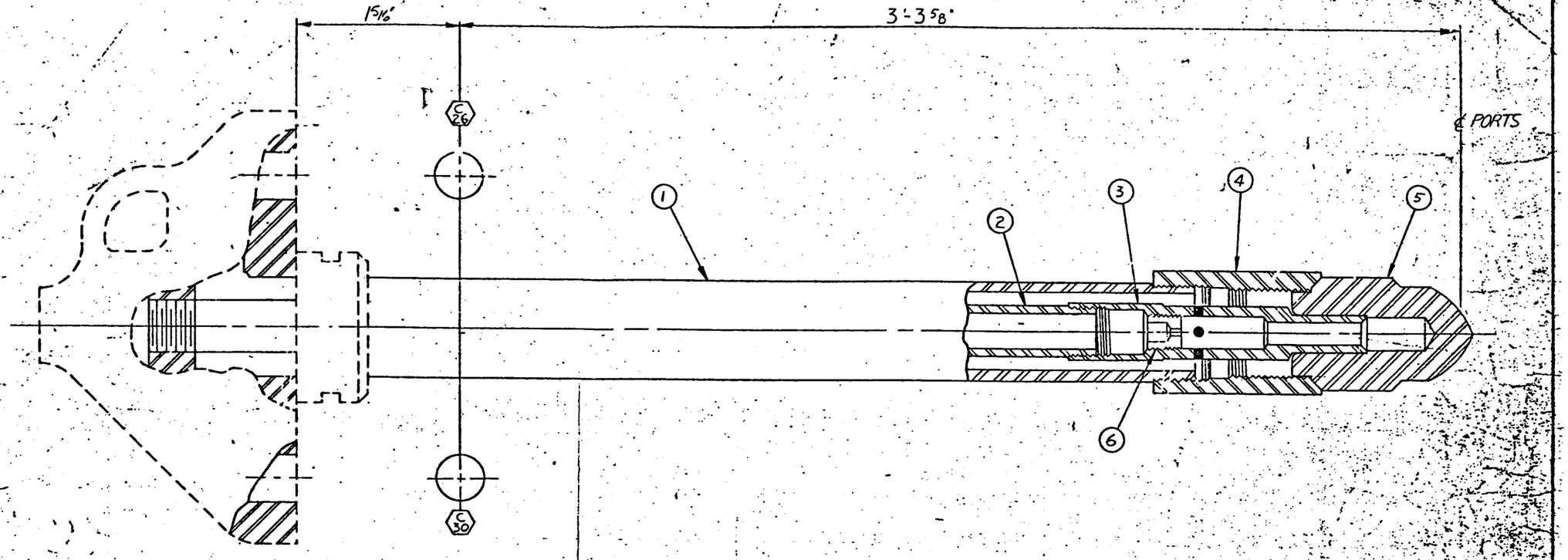
ITEM	QTY.	DESCRIPTION	PART NO.	MAT'L.
1	1	O-RING (1⅜" I.D. x 2⅝" O.D.)		BUNA "N"
2	1	STEAM TUBE	1" PIPE	HAST. C
3	1	WASTE TUBE	⅜" PIPE	HAST. C
4	1	SLEEVE	DH SLV	HAST. C
5	1	MEMBER	DH-1	HAST. C
6	1	DH-WASTE TIP	DH TIP	HAST. C

USER: MONSANTO
JOBSITE: ST. LOUIS, MO.
S.O. No. 081181
P.O. No. 3008-9-0037

JOHN ZINK COMPANY
TULSA
DH-WASTE GUN INSERT
GUN #4
FOR: MONSANTO

DR. JMT	DATE 3-17-76	CERTIFIED	DRAWING NUMBER A-0-81181-607
CK. RN	DATE 3-19-76	McG.H	SCALE
APP. [illegible]	DATE 3-22-76	DATE 3-22-76	

REV.			
1	ADDED MK NO.	3-22-76	JMT



PART SECTIONAL ELEVATION
ONE - M: A-605-1

REFERENCE DWG'S.
B-O-81181-601 : 'HI'-24 BURNER ASSY.

PARTS LIST				
ITEM	QTY.	DESCRIPTION	PART #	MATL
1	1	STEAM TUBE 1" SCH 40 PIPE	—	HAST.C
2	1	OIL TUBE 3/8 SCH. 40 PIPE	—	A-120
3	1	EA ATOMIZER	EA-7	303 ss
4	1	SLEEVE	#225	HAST.C
5	1	EA TIP	EA-765	HAST.C
6	1	EA WASTE SPUD	PAT-953-7	HAST.C

MONSANTO COMPANY Order No. 3008-9-0037
CORPORATE ENGR. DEPT. Project (CEA) No. 3008
Location St. Louis, Mo. Area 02
Equipment No. 111.000 ; 111.000 ; 169.000
Description Vaporizer / Scrubber System
Reviewed by Jeff Dolstein Date 3-25-76
☐ Make Corrections noted ☐ Leave as Shown ☒ Final

REV.		
REV.		
REV.		
REV. 1	ADDED MK NO PER ECN #1266	DLH 3-3-76

USER: MONSANTO

JOHN ZINK COMPANY
TULSA OKLAHOMA

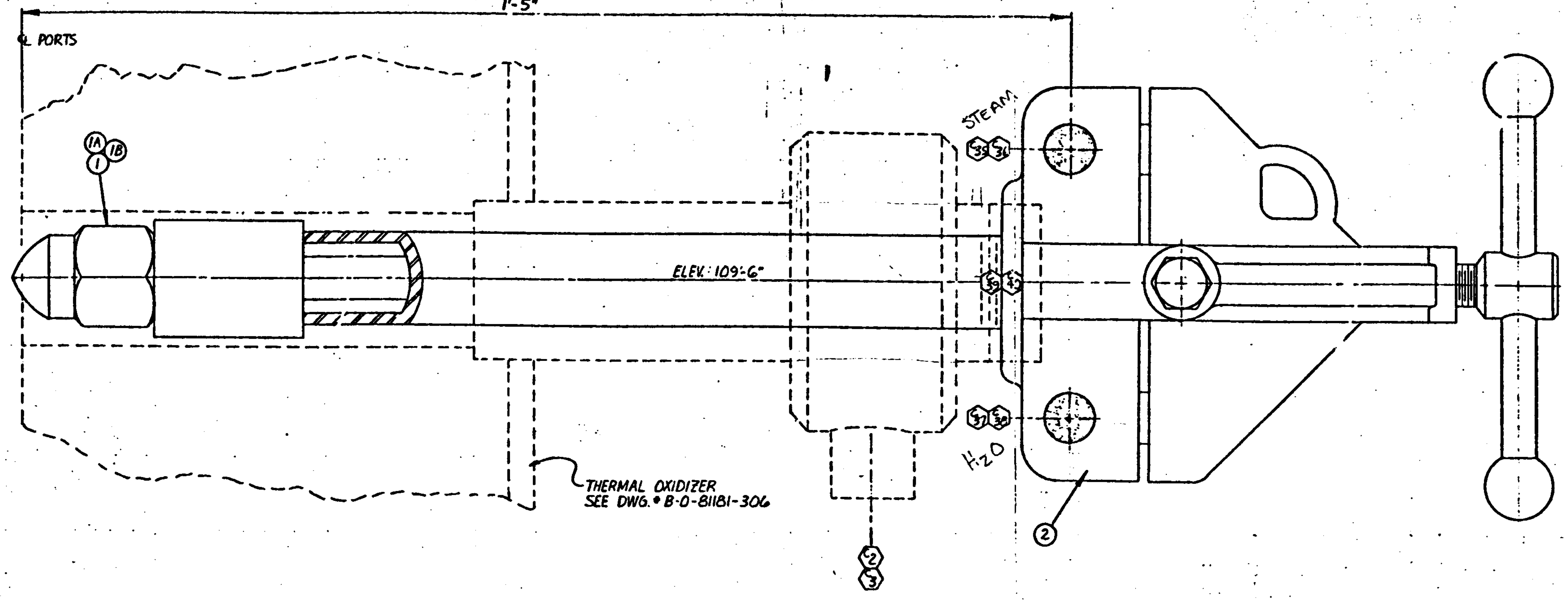
JOBSITE: ST. LOUIS, MO.

'EA' OIL GUN INSERT
GUN #1

S.O.NO. 081181

P.O.NO. 3008-9-0037 FOR: MONSANTO

DR. DLH	DATE 1-12-76	CERTIFIED	DRAWING NUMBER A-Q-81181-605
CK. RJ	DATE 1-19-76		
APP. GF	DATE 1-20-76	DATE 2-23-76	SCALE



PART SECTIONAL ELEVATION

TWO-MK-B-662-1

PARTS LIST (PER GUN)

ITEM	QUAN.	DESCRIPTION	PART NO.	MATERIAL
1	1	'EA' GUN INSERT / A-ST-0369-1	—	—
1A	1	'EA' TIP	EA-10A	416 SS
1B	1	'EA' ATOMIZER	EA-10	303 SS
2	1	OIL BODY / CA-ST-0027-2	RZ-56-2	DUCTILE

NOZZLE LEGEND

ITEM	QTY.	DESCRIPTION
C3,C3	2	3/4" PURGE AIR CONN. (3/4"-N.P.T. FEMALE)
C35,36	2	1/2" TEMPERING STEAM CONN. (1/2"-N.P.T. FEMALE)
C37,38	2	1/2" TEMPERING WATER CONN. (1/2"-N.P.T. FEMALE)
C39,40	2	2" TEMPERING GUN CONN. (2"-N.P.T. MALE)

GENERAL NOTES

1. FINISH EXTERIOR: PAINT W/ ONE COAT OF HIGH TEMPERATURE ALUMINUM (C.S. SURFACES ONLY)
2. ALL MK. NO'S TO BE PRECEDED BY S.O. NO.

REFERENCE DRAWINGS

B-0-81181-306 : THERMAL OXIDIZER

A-ST-0369-1 : 'EA' GUN ASSY.

RELEASE DATA

FLOW RATE: 4 G.P.M. MAX. @ 65 PSIG of TEMPERING WATER AND 100 PSIG STEAM

MONSANTO COMPANY
CORPORATE ENGR. DEPT. Project (CEA) No. 3008
Equipment No. 111.000 : 114.000 : 169.000
Date 3-3-76

REV. NO. 4

REV. NO. 3

REV. NO. 2

REV. NO. 1 ADDED MK NO'S. PER ECN #1266 3-3-76 JWT

USER: MONSANTO

JOHN ZINK COMPANY
TULSA OKLAHOMA

JOBSITE: ST. LOUIS, MO.

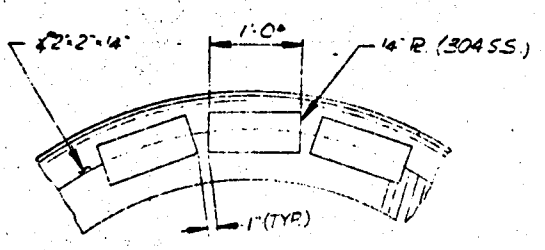
TEMPERING GUN

S.O. NO. 081181

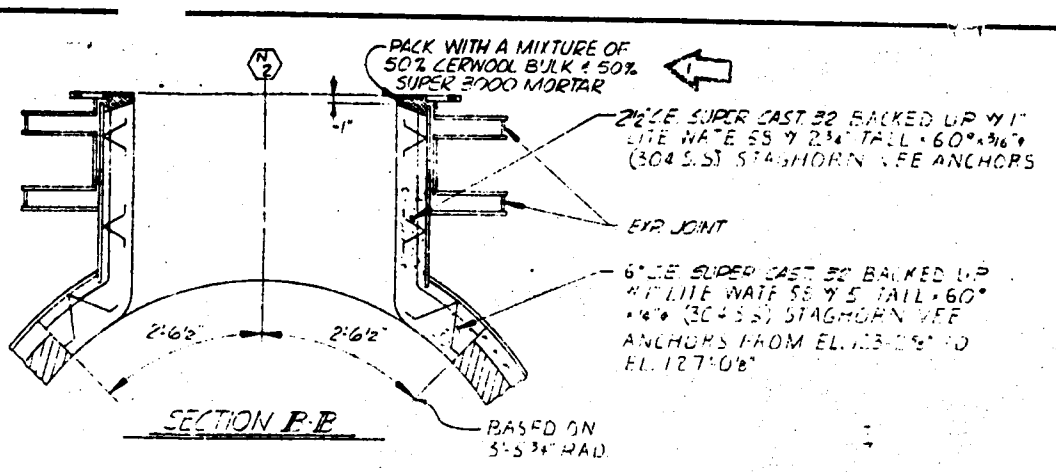
P.O. NO. 3008-9-0037

FOR: MONSANTO

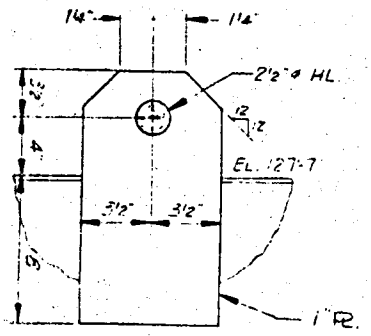
DR. JW	DATE 1-6-76	CERTIFIED M.C.P.	DRAWING NUMBER B-0-81181-602	REV 1
CK. RN	DATE 1-19-76		SCALE	
APP. BJ	DATE 1-20-76	DATE 3-3-76		



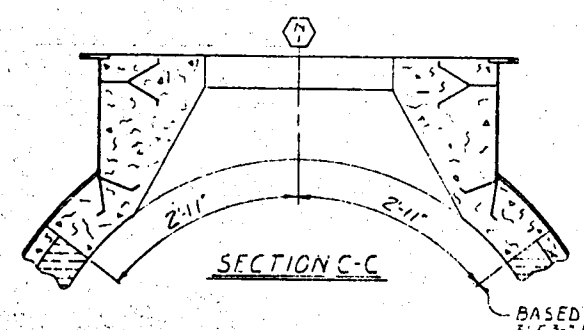
SECTION A-A
(REFRACTORY SUPPORT SHELF)



SECTION B-B



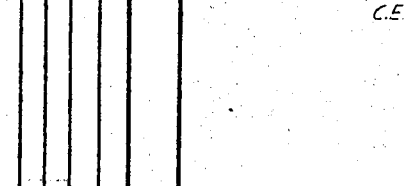
LIFTING LUG DETAIL
(2 REQ'D., 180° APART)



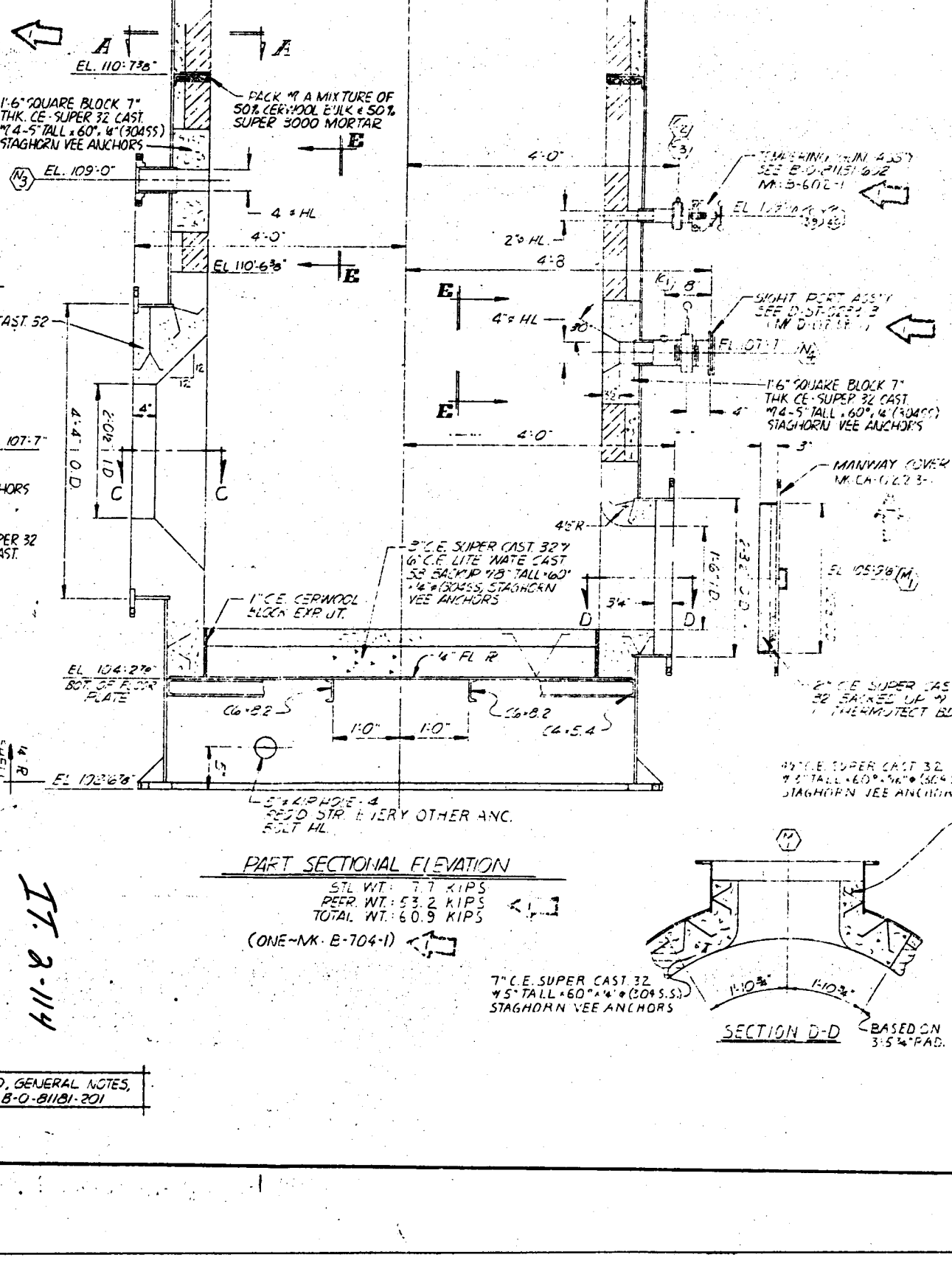
SECTION C-C

REFRACTORY QUANTITIES

QTY.	DESCRIPTION	% OVER
11,020#	SUPER CAST 32	30%
8,234#	LITE WATE 58 CAST.	25%
930	MULLITEX HB F.B. 9"×4½"×3" # 2 ARCH	2%
1643	MULLITEX HB F.B. 9"×4½"×3" # 1 ARCH	2%
1080#	CHEMAL 111 MORTAR	5%
66 FT.	CERWOOL BLOCK - 1" THK × 1'-0" × 3'-0"	5%
15 Cu.Ft.	CERWOOL "BULK A"	5%
4 #	THERMOTECT BOARD - 1" THK.	5%
144#	SUPER 3000 MORTAR	5%



TYPICAL ANCHOR SPACING



PART SECTIONAL ELEVATION

STL WT.: 7.7 KIPS
PEER WT.: 53.2 KIPS
TOTAL WT.: 60.9 KIPS
(ONE-MK. B-704-1)

SECTION D-D

NOTE: FOR NOZZLE LEGEND, GENERAL NOTES, & REFERENCE DWG'S, SEE B-0-81181-201

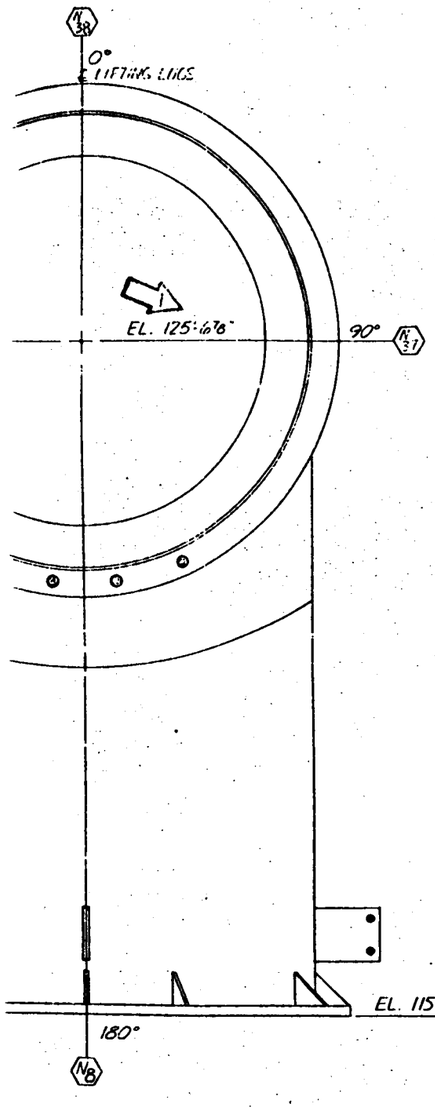
IT. 2-114
893.62
Equip #23-2054

NO.	REVISION DESCRIPTION	BY	CK.	APP.	DATE
1	REV. PER CUST. REQ. ECN #1266	DLH	DAVE	RJ	2-13-76

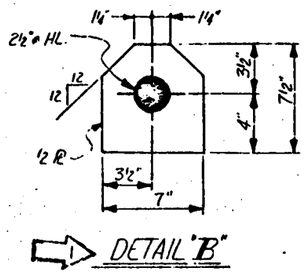
THIS DRAWING AND THE INFORMATION REVEALED BY IT IS SUBMITTED WITH THE UNDERSTANDING THAT IT IS THE CONFIDENTIAL AND PROPRIETORY PROPERTY OF JOHN ZINK CO. AND IS NOT TO BE DISCLOSED TO OTHERS. ALL DOCUMENTS MUST BE RETURNED UPON REQUEST. UNAUTHORIZED USE IS EXPRESSLY FORBIDDEN.

USER: MONSANTO
JOBSITE: ST. LOUIS, MO.
S.O. NO. 081181
P.O. NO. 3008-9-0037
DR. DAVE
CK. RJ
APP. DATE 2-13-76
APP. DATE 2-19-76
APP. DATE 1-19-76
APP. DATE 1-20-76
CERTIFIED DATE 7-23-76

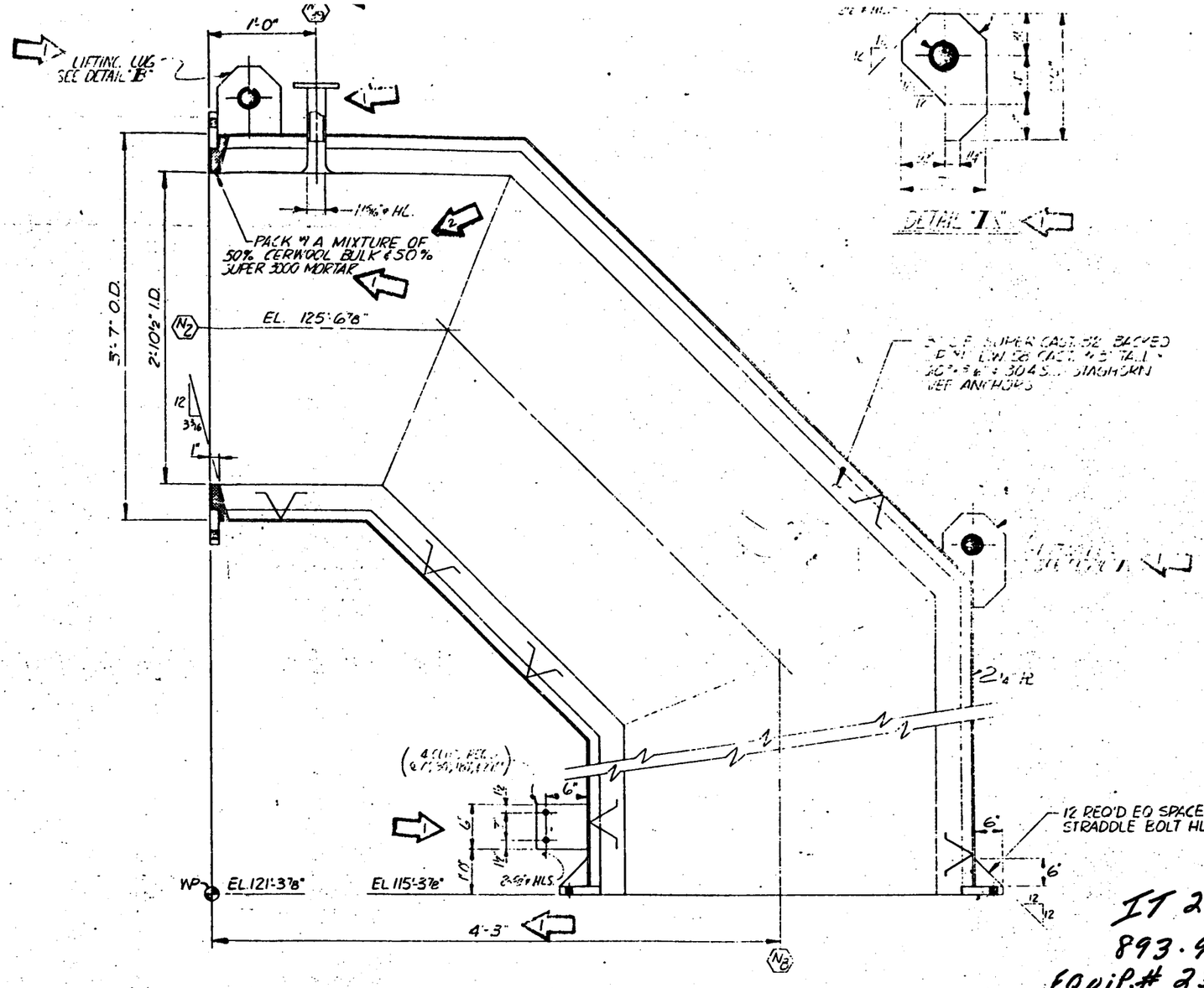
JOHN ZINK COMPANY
TULSA OKLAHOMA
THERMAL OXIDIZER REFRACTORY DET.
FOR: MONSANTO
SCALE 1"=1'-0"
DRAWING NUMBER B-0-81181-306
REV. 1



END VIEW
(TRUE ORIENTATION)



1 DETAIL "B"



PART SECTIONAL ELEVATION

1

STL. WT.	1.4 KIPS
REFR. WT.	5.8 KIPS
TOTAL WT.	7.2 KIPS

ONE-MK: B-702-1

NOTE: FOR NOZZLE LEGEND, GENERAL NOTES, & REFERENCE DWG'S., SEE B-C-81181-201

IT 2-114
893.92
EQUIP.# 23-2054

REFRACTORY QUANTITIES

QTY.	DESCRIPTION	% OVER
833#	C.E. LITE WATE 58 CAST.	25%
5470#	C.E. SUPER CAST. 32	30%
½ FT³	C.E. CERWOOL BULK 'A'	5%
25#	MORTAR - SUPER 3000	5%

1 MK: B-201-9
2 MK: B-201-2

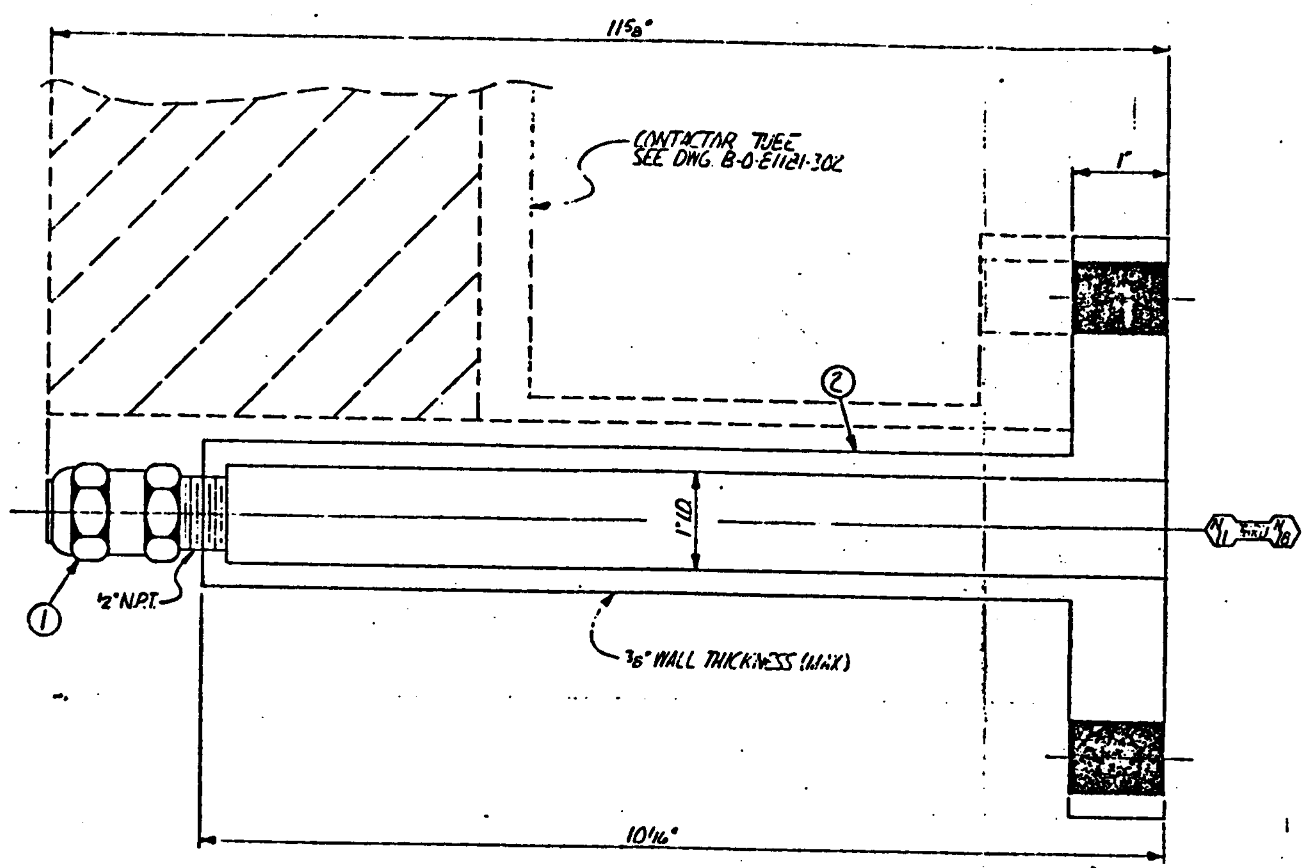
NO.	REVISION DESCRIPTION	BY	CK.	APP.	DATE
2	REVISED EXP. JT. CALL OUT	DAVE	dk	DE	3-29-76
1	REVISED PER CUSTOMER REQUEST ECN # 1266	MAB	dh	EP	2-13-76

USER: MONSANTO
JOBSITE: ST. LOUIS, MO.
S.O. NO. QB1181
P.O. NO. 3008-9-0037

JOHN ZINK COMPANY
TULSA OKLAHOMA

ERECHING DUCT
REFRACTORY DETAIL
FOR: MONSANTO

DR. DAVE	DATE 12-15-75	CERTIFIED	DRAWING NUMBER B-C-81181-304 REV 2
CK. RJ	DATE 1-19-76		
APP. EP	DATE 1-20-76	DATE 3-22-76	SCALE ¾"



PART SECTIONAL ELEVATION
B-REJO-MK: B-606-1

PARTS LIST (PER GUN)

ITEM	QUAN.	DESCRIPTION	PART NO.	MAT'L.
1	1	SPRAYING SYSTEMS TIP	1/2 GG 32	TITANIUM
2	1	QUENCH GUN	—	TEFLON

REFERENCE DWG'S

B-0-E1181-302 : CONTACTOR TUBE DETAILS

GENERAL NOTES

1. ALL BOLT HOLES TO STRADDLE NORMAL ℄'s
2. ALL MK-NO'S TO BE PRECEDED BY S.O. NO.
3. FINISH: NONE REQUIRED

FLOW RATE: 7.4 G.P.M.

NOZZLE LEGEND

ITEM	QUAN.	DESCRIPTION
N11-18	8	2" QUENCH GUN CONN. - (2"-150# F.F.)

MONSANTO COMPANY Order No. 3005-5-0237
CORPORATE ENGR. DEPT. Noted (QA) No. 3009
Location St. Louis Area
Equipment No. 111 000 - 000 161 000
Description
Reviewed By
☐ Make Corrections noted ☐ Leave as Shown ☒ Final

REV. NO. 4
REV. NO. 3
REV. NO. 2
REV. NO. 1

USER: MONSANTO

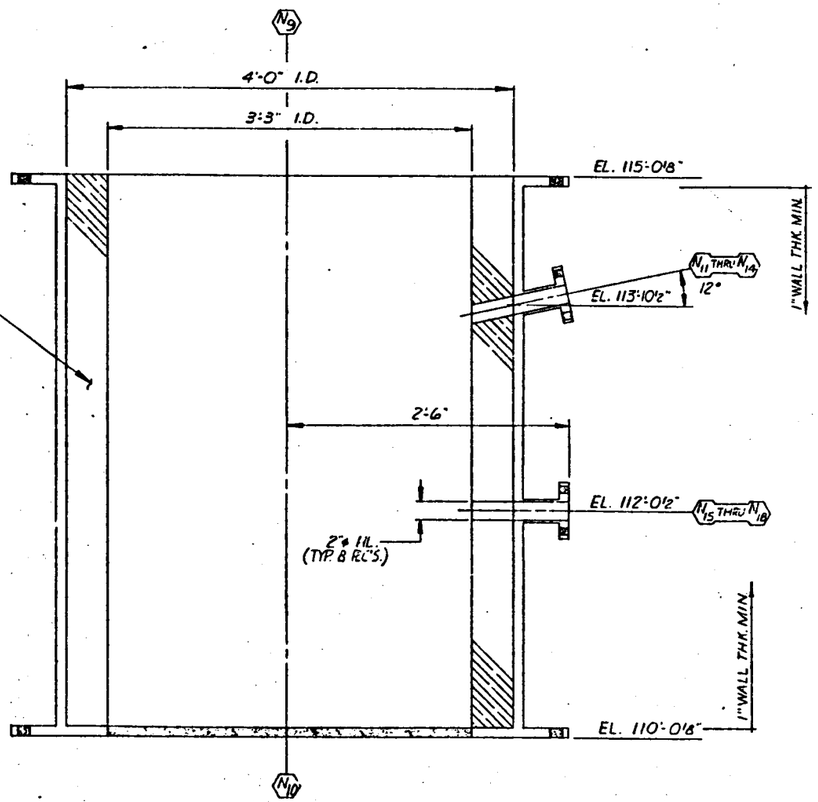
JOHN ZINK COMPANY
TULSA OKLAHOMA

JOBSITE: ST. LOUIS, MISSOURI

QUENCH GUN ASSY

S.O. NO. 11181
P.O. NO. 3CA-9-0137
FOR: MONSANTO

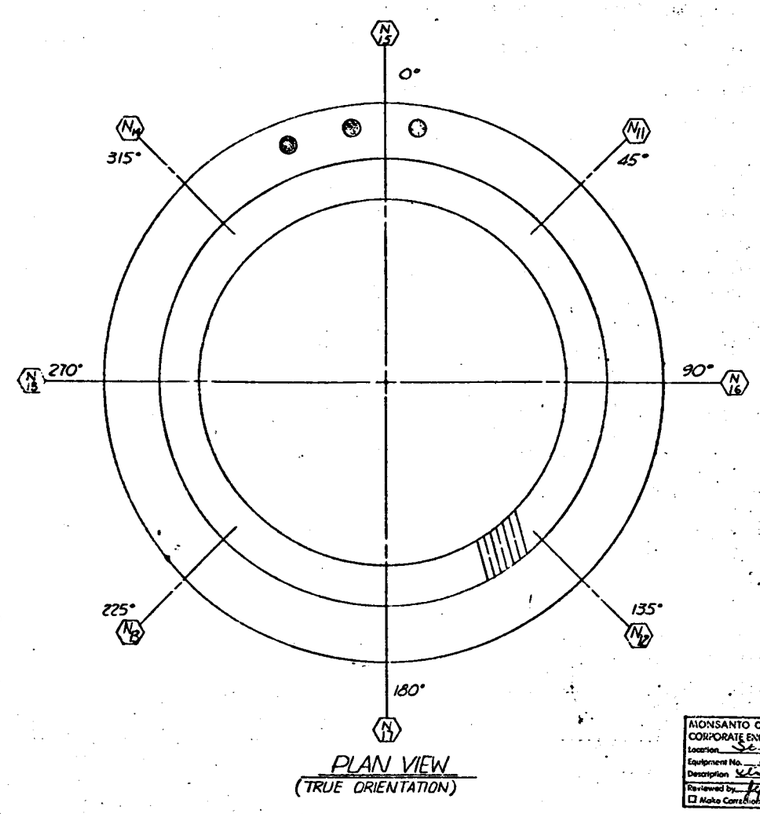
DR.	DATE	CERTIFIED	DRAWING NUMBER	REV
CK. dh	DATE 2-8-76		11181-606	
APP.	DATE 3-22-76	DATE	SCALE	



PART SECTIONAL ELEV.

REFR. WT. = 3.2 KIPS

ONE-MK:B-302-1



PLAN VIEW

(TRUE ORIENTATION)

MONSANTO COMPANY	Order No. 3003-5-0037
CORPORATE ENGR. DEPT.	Project (CEA) No. 3008
Location St. Louis, Mo.	Area 02
Equipment No. 111.000, 114.000, 145.000	
Description Ammonium Sulfate System	
Reviewed by: [illegible]	Date 3-25-76
☐ Make Corrections noted	☐ Leave as Shown

X Print

NOTE: FOR NOZZLE LEGEND GENERAL NOTES, & REFERENCE DWG'S., SEE B-D-81181-201

MATERIALS OF CONSTRUCTION TO BE FE.P. TEFLON LINED (90 MILS. THICKNESS) GLASS FIBER REINFORCED DERAKANE 410-45 VINYL ESTER RESIN CONSTRUCTION

NOTE: TO BE CONSTRUCTED PER IL STD. SPEC. #3-1-1 P12 & 14.

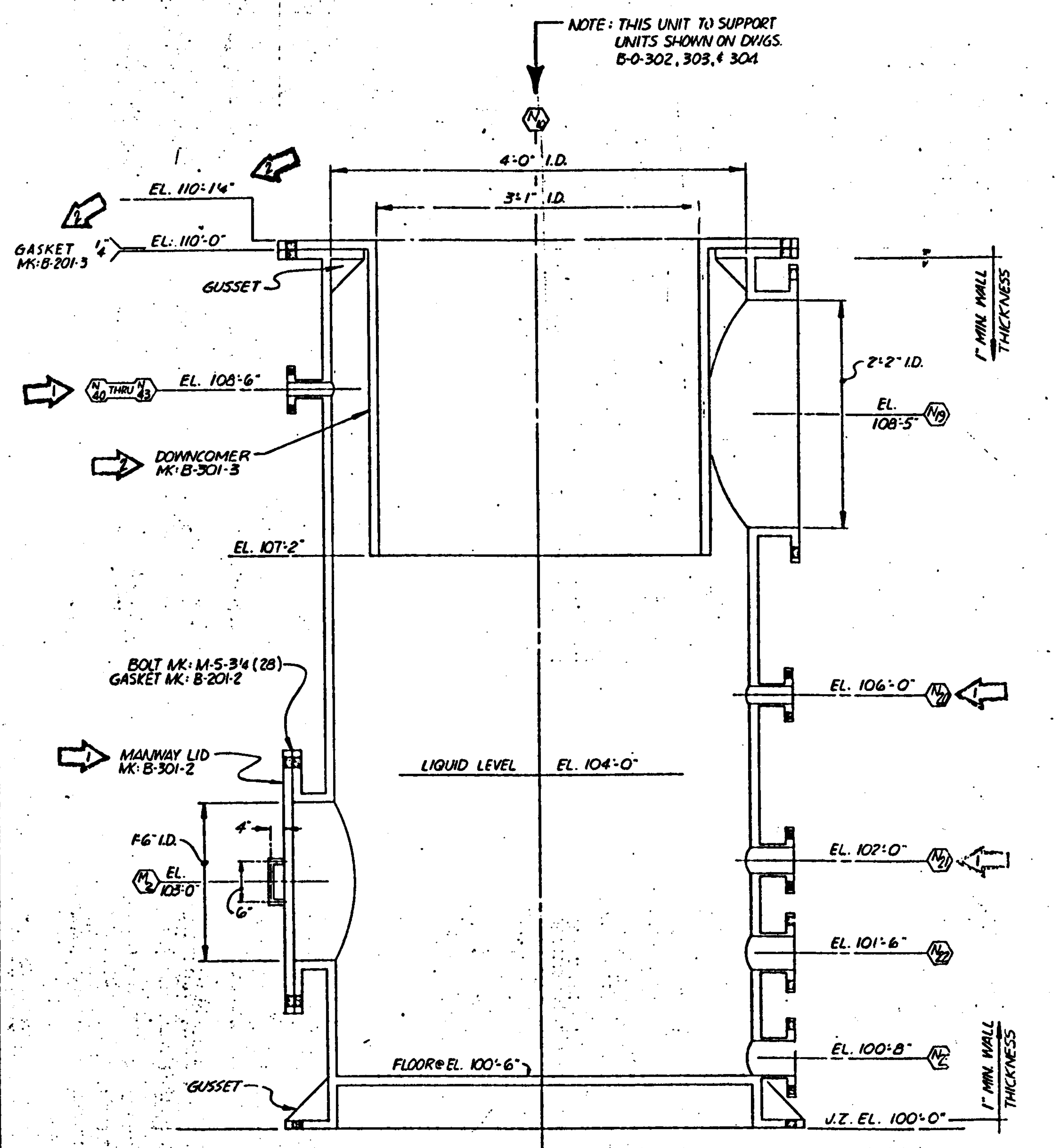
REFRACTORY QUANTITIES

QTY.	DESCRIPTION	% OVER
43	C.E. MULLITEX F.B. 9"x4½"x3" #3 ARCH	2%
522	C.E. MULLITEX F.B. 9"x4½"x3" #2 ARCH	2%

MK:B-201-7
MK:B-201-8

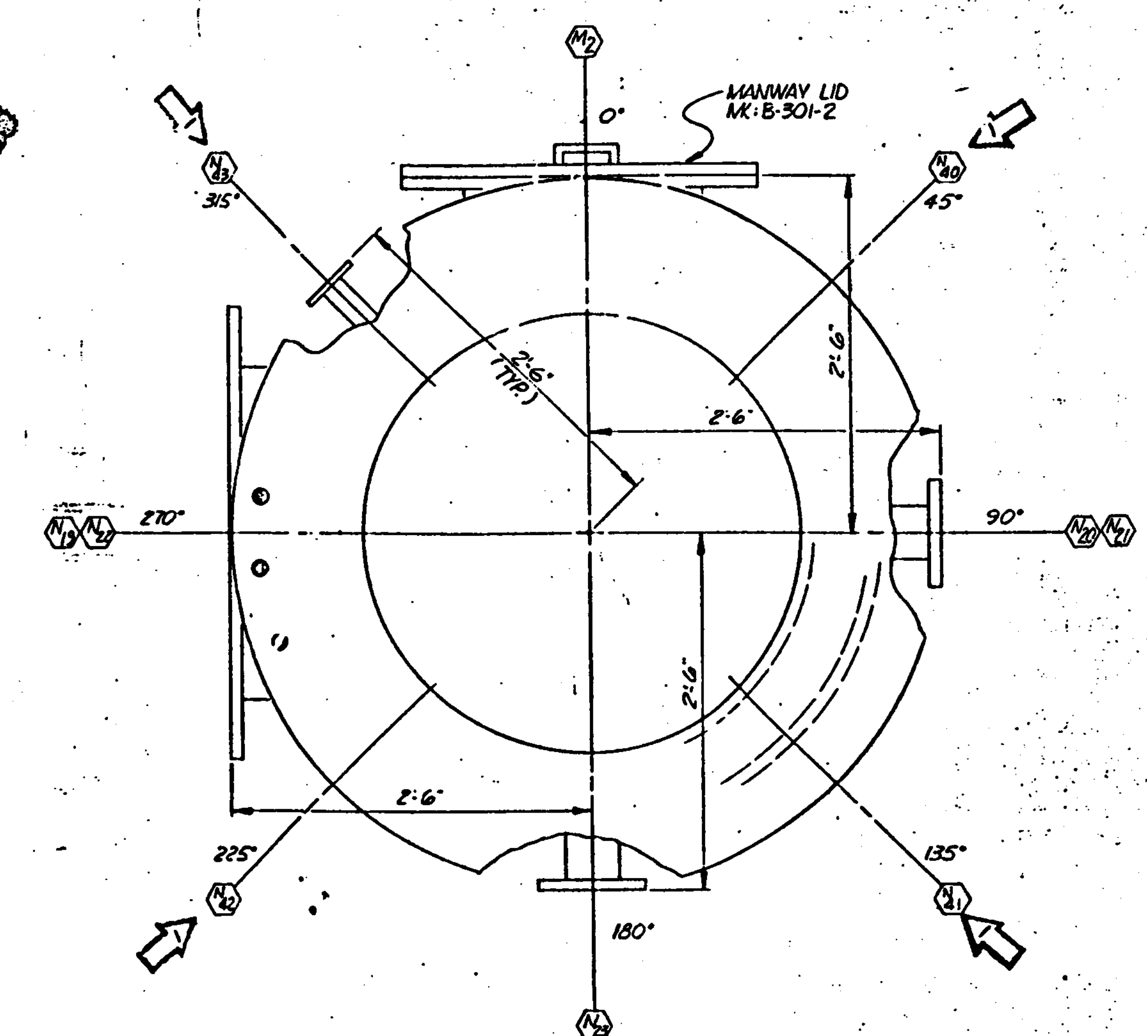
NO	REVISION DESCRIPTION	BY	CK.	APP.	DATE
1	ADDED MK. NO'S. (ECN #1766)	JMT	dh	JDP	2-13-76

USER: MONSANTO	JOHN ZINK COMPANY TULSA OKLAHOMA
JOBSITE: ST. LOUIS, MO.	CONTACTOR TUBE DET.
S.O. NO. 081181	
P.O. NO. 3003-5-0037	FOR: MONSANTO
DR. DATE	DATE 12-12-75
CK. RN	DATE 1-17-76
APP. BJ	DATE 1-20-76
CERTIFIED	DATE 3-25-76
SCALE	DRAWING NUMBER B-D-81181-302 REV 1



PART SECTIONAL ELEVATION

ONE ~ MK: B-301-1



PLAN VIEW
(TRUE ORIENTATION)

NOTE: TO BE CONSTRUCTED PER JZ STD. SPEC. #3-1-1 P.12 & 14.

NOTE: FOR NOZZLE LEGEND, GENERAL NOTES, & REFERENCE DWG'S, SEE B-0-81181-201.

NOTE: MAT'LS. OF CONSTRUCTION TO BE F.E.P. TEFLON LINED (30 MILS THK.) GLASS FIBER REINFORCED DERAKANE 470-45 VINYL ESTER RESIN CONSTRUCTION.

MONSANTO COMPANY CORPORATE ENGR. DEPT. Order No. 3008-9-0037 Location St. Louis, Mo. Equipment No. 110000-111000-169000 Reviewed by — Final

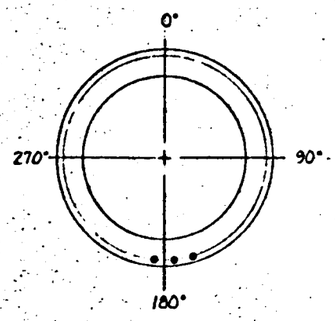
NO.	REVISION DESCRIPTION	BY	CK.	APP.	DATE
2	REV. PER ECN # 2184, CHG'D GASKET THK.	DAVE	RJ	DP	3-25-76
1	REVISED PER CUST. REQUEST & ECN # 1266	JMT	dh	DP	2-13-76

USER: MONSANTO
JOBSITE: ST. LOUIS, MO.
S.O. NO. 0-81181
P.O. NO. 3008-9-0037
DR. DAVE — DATE 12-12-75
CK. RJ — DATE 1-19-76
APP. EJ — DATE 1-20-76

JOHN ZINK COMPANY
TULSA OKLAHOMA

QUENCH POT DETAIL

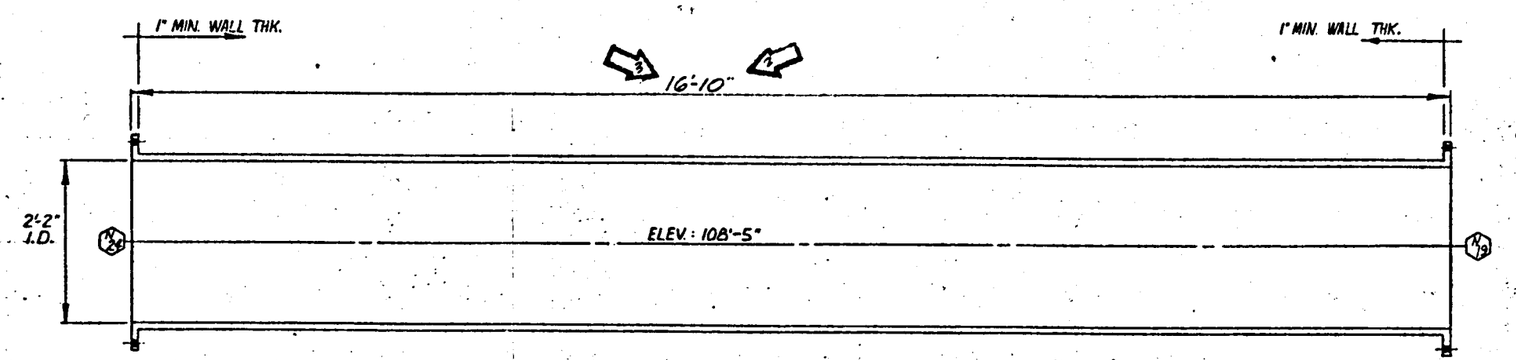
FOR: MONSANTO
CERTIFIED — DATE 3-22-76
DRAWING NUMBER B-0-81181-301 — REV 2
SCALE —



END VIEW

NOTE: FOR NOZZLE LEGEND, GENERAL NOTES AND REFERENCE DRAWINGS SEE B-0-81181-201

NOTE: TO BE CONSTRUCTED PER JZ STD. SPEC. # 3-1-1, P.12 & 14.



SIDE SECTIONAL ELEVATION

ONE-MK: B-909-1

MATERIALS OF CONSTRUCTION TO BE FEP TEFLON LINED (90 MILS THICKNESS) GLASS FIBER REINFORCED DERAKANE 470-45 VINYL ESTER RESIN CONSTRUCTION

MONSANTO COMPANY Order No. 3C09-6607
CORPORATE ENGR. DEPT. Project (CEA) No. 3C09
Location St. Louis, Mo. Area
Equipment No.
Description
Contractor JZ Date 6-1-76
☐ As-Built Corrections noted ☐ Errors as Shown ☒

THIS DRAWING AND THE INFORMATION REVEALED BY IT IS SUBMITTED WITH THE UNDERSTANDING THAT IT IS THE CONFIDENTIAL AND PROPRIETARY PROPERTY OF JOHN ZINK CO. AND IS NOT TO BE DISCLOSED TO OTHERS WITHOUT WRITTEN PERMISSION. UNAUTHORIZED USE IS EXPRESSLY FORBIDDEN. ALL DOCUMENTS MUST BE RETURNED UPON REQUEST

NO.	REVISION DESCRIPTION	BY	CK.	APP.	DATE
3	REV. LGTH. /ECN # 2197	JH	[illegible]	TJ	5-25-76
2	REV. PER ECN # 2184, REV. LENGTH	DAVE	KRJ	DJP	3-23-76
1	ADDED MK NO (ECN # 1966)	JMTT	JH	DJP	2-13-76

USER: MONSANTO
JOBSITE: ST. LOUIS, MO.
S.O. NO. 061181
P.O. NO. 3C09-3-0-7
DR. JV DATE 12-30-75
CK. KRJ DATE [illegible]
APP. EZ DATE 1/22/76

JOHN ZINK COMPANY
TULSA OKLAHOMA

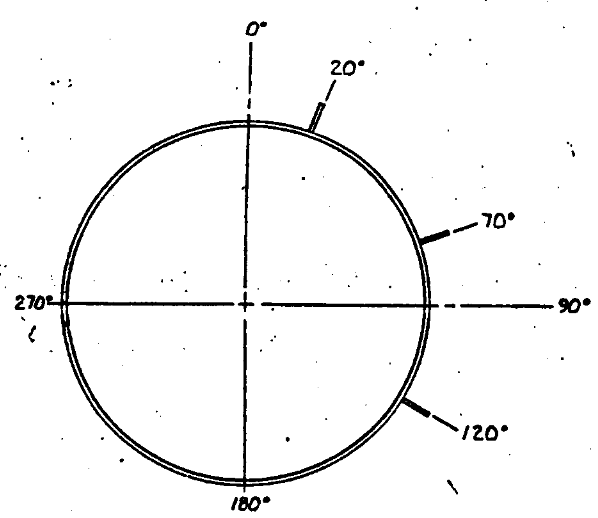
CONNECTING DUCT

FOR: MONSANTO

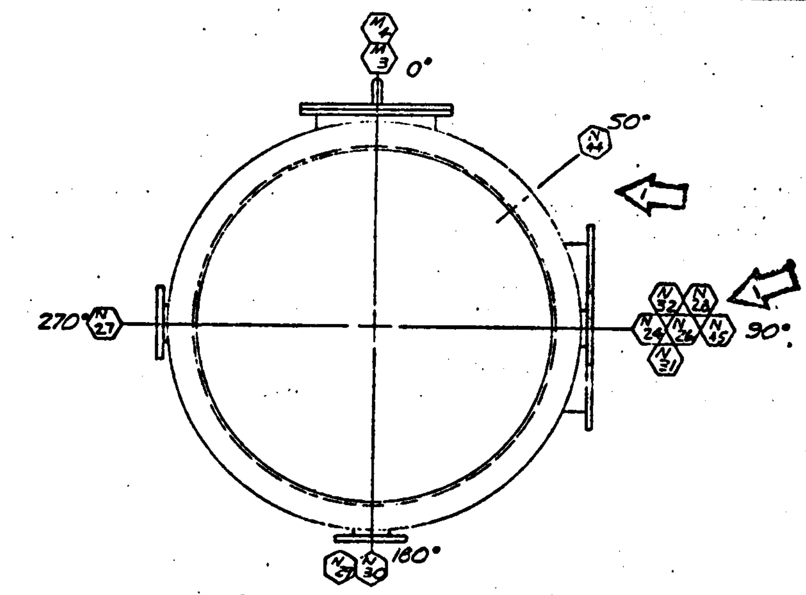
CERTIFIED
DATE 2-23-76

DRAWING NUMBER B-0-81181-309 REV 3

SCALE 3/4



SECTION A
(TRUE ORIENTATION)

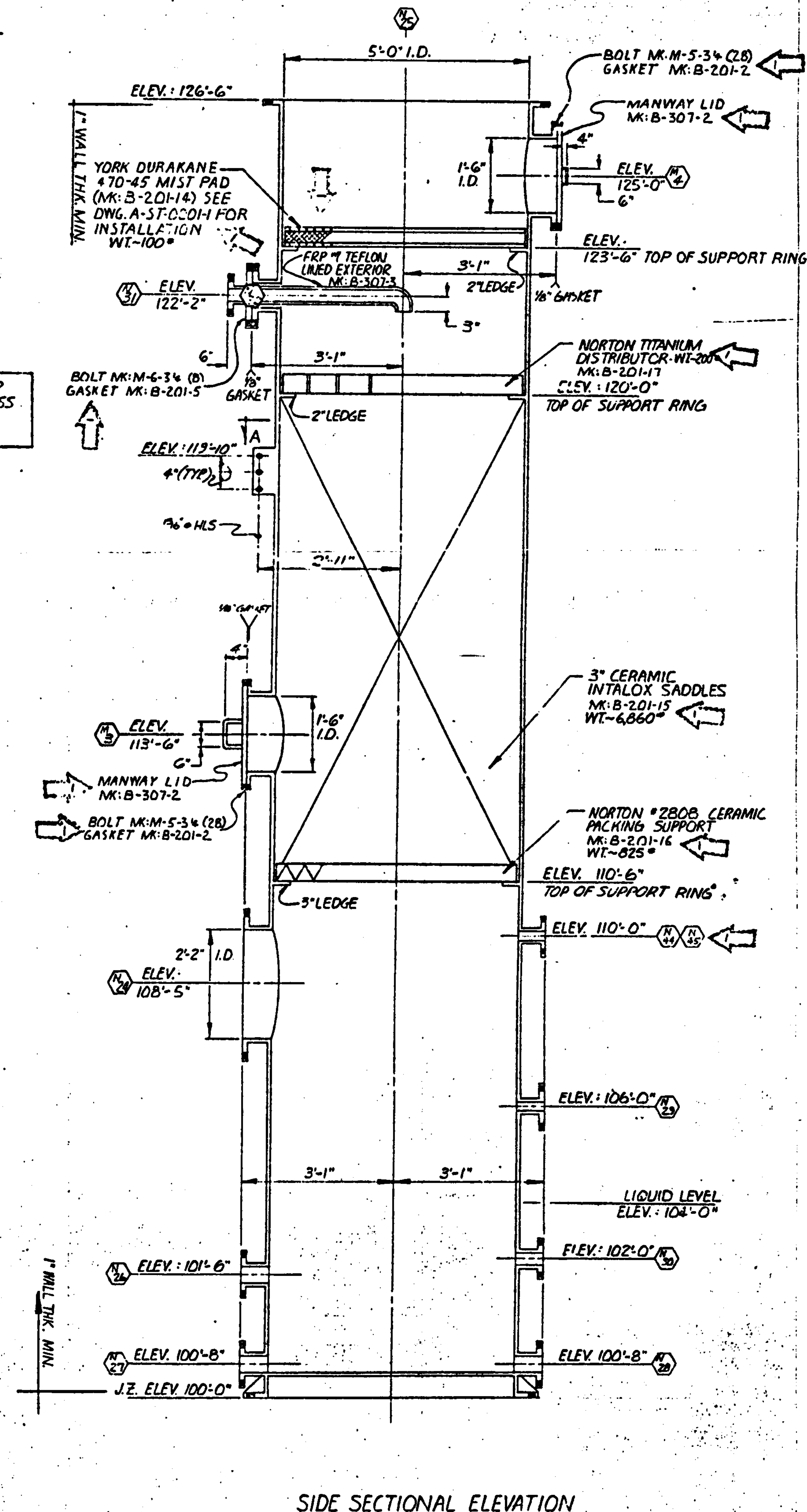


PLAN VIEW
(TRUE ORIENTATION)

NOTE: FOR NOZZLE LEGEND, GENERAL NOTES, AND REFERENCE DRAWINGS SEE B-0-81131-201

NOTE: TO BE CONSTRUCTED PER JZ STD. SPEC. #3-1-1, P.12 & 14.

MATERIALS OF CONSTRUCTION TO BE FEP TEFLON LINED (90MILS MIN. THICKNESS) GLASS FIBER REINFORCED DERAKANE 470.45 VINYL ESTER RESIN CONSTRUCTION.



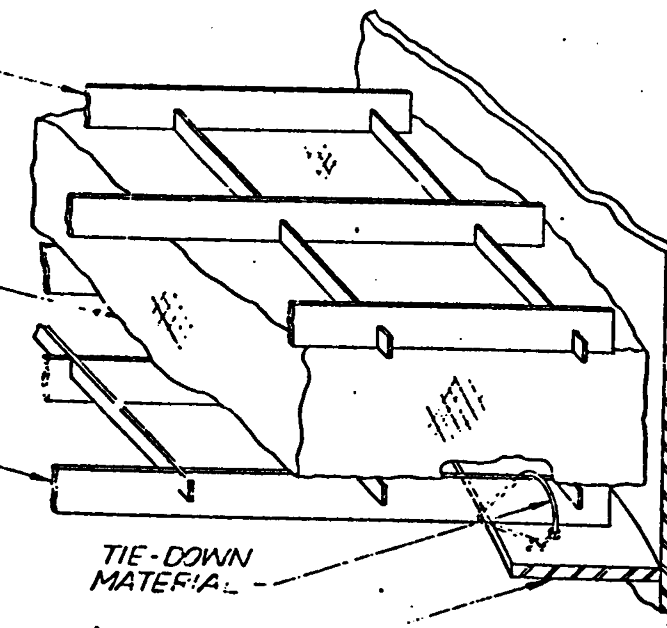
SIDE SECTIONAL ELEVATION
(ONE-MK: B-307-1)

NO.	REVISION DESCRIPTION	BY	CK.	APP.	DATE
1	REV. PER CUST. REQ. ECN #1266 & #1254	C.H	dh	EP	2-13-76

JOHN ZINK COMPANY
TULSA OKLAHOMA

USER: MONSANTO
JOBSITE: ST. LOUIS, MO.
ABSORBER DETAIL (LOWER SECTION)
S.O. NO. 081131
F.O. NO. 3008-3-66371
FOR: MONSANTO
DR. JW DATE 12-23-75
CK. RJ DATE 1-14-76
APP. OJ DATE 1-20-76
CERTIFIED DATE 3-23-76
DRAWING NUMBER B-0-81131-307
REV 1
SCALE

MONSANTO COMPANY
CORPORATE ENGR. DEPT.
Order No. 3008-3-66371



CUSTOMER NOTE:
MIST PAD ASS'Y TO BE INSTALLED AS SHOWN. TIE BOTTOM GRID TO SUPPORT RING USING MAT'L. SUPPLIED W/ ASSY. HOLES IN SUPPORT RING ON APPROXIMATELY 2'0" CENTERS.

MONSANTO COMPANY Order No. 3008-4-0037
CORPORATE ENGR. DEPT. Project (CEA) No. 3008
Location St. Louis Mo. Area C2
Equipment No. 114.CCO
Description
Reviewed by [illegible] Date 5-2?-76
☐ Make Corrections noted ☐ Leave as shown

NO.	REVISION DESCRIPTION	BY	CK	APP	DATE
2	REDRAWN	RN	dh		1-26-76

USER:
JOBSITE:
S.O. NO.
P.O. NO.

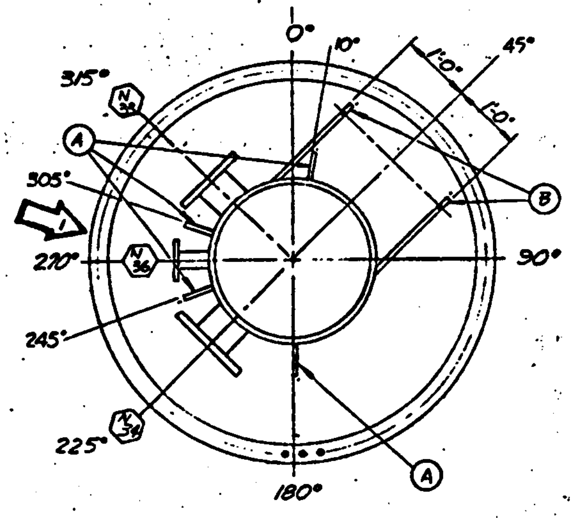
JOHN ZINK COMPANY
TULSA OKLAHOMA

MIST PAD
INSTALLATION DETAILS

FOR: STANDARD

DR RN	DATE 1-26-76	CERTIFIED	DRAWING NUMBER A-ST-0001
CK. dh	DATE 1-26-76		REV 2
APP.	DATE	DATE	SCALE

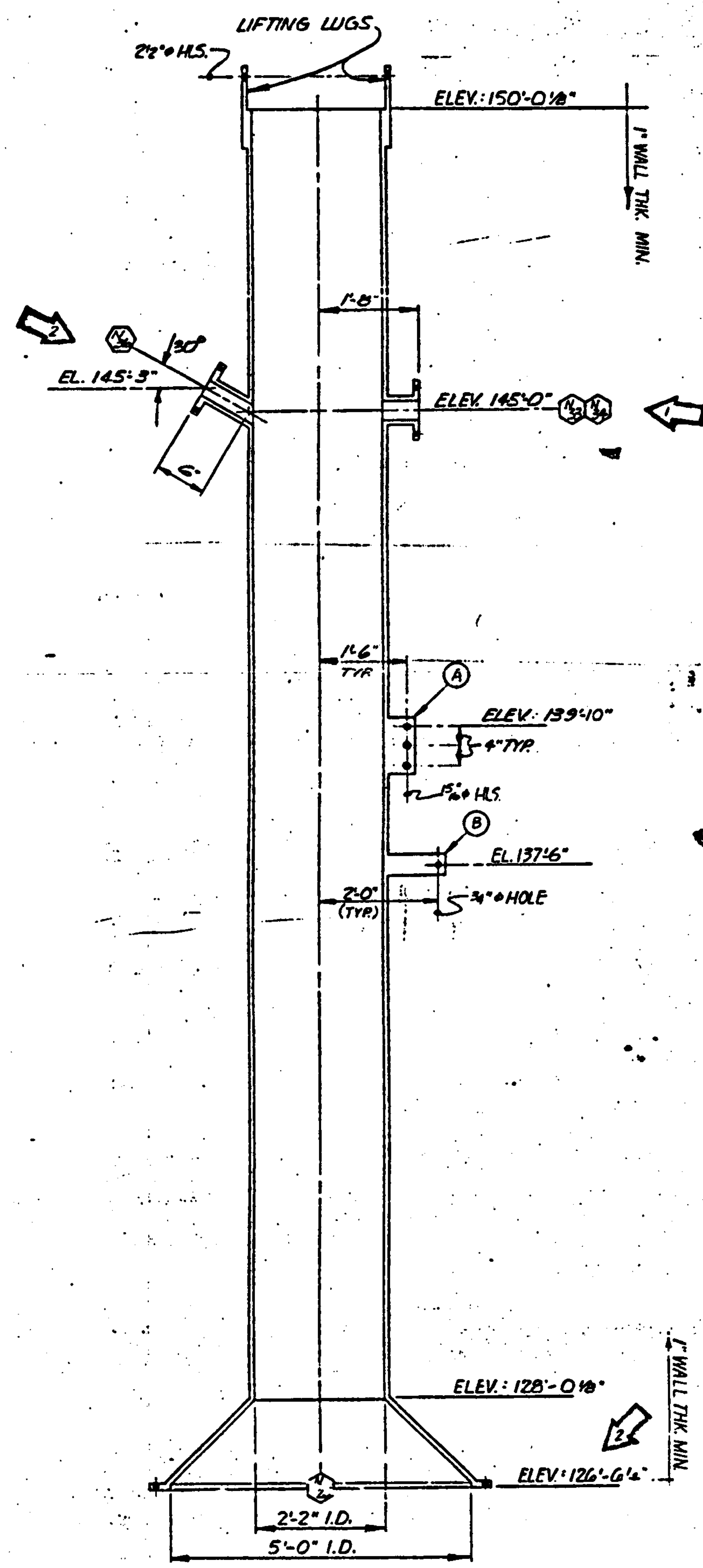
NOTE: FOR NOZZLE LEGEND, GENERAL NOTES, AND REFERENCE DRAWINGS SEE B-0-81181-201



PLAN VIEW
TRUE ORIENTATION

MATERIALS OF CONSTRUCTION TO BE FEP TEFLON LINED (90 MILS THICKNESS) GLASS FIBER REINFORCED DERAKANE 470-45 VINYL ESTER RESIN CONSTRUCTION

NOTE: TO BE CONSTRUCTED PER JZ STD. SPEC. #3-1-1, P.12 & 14.



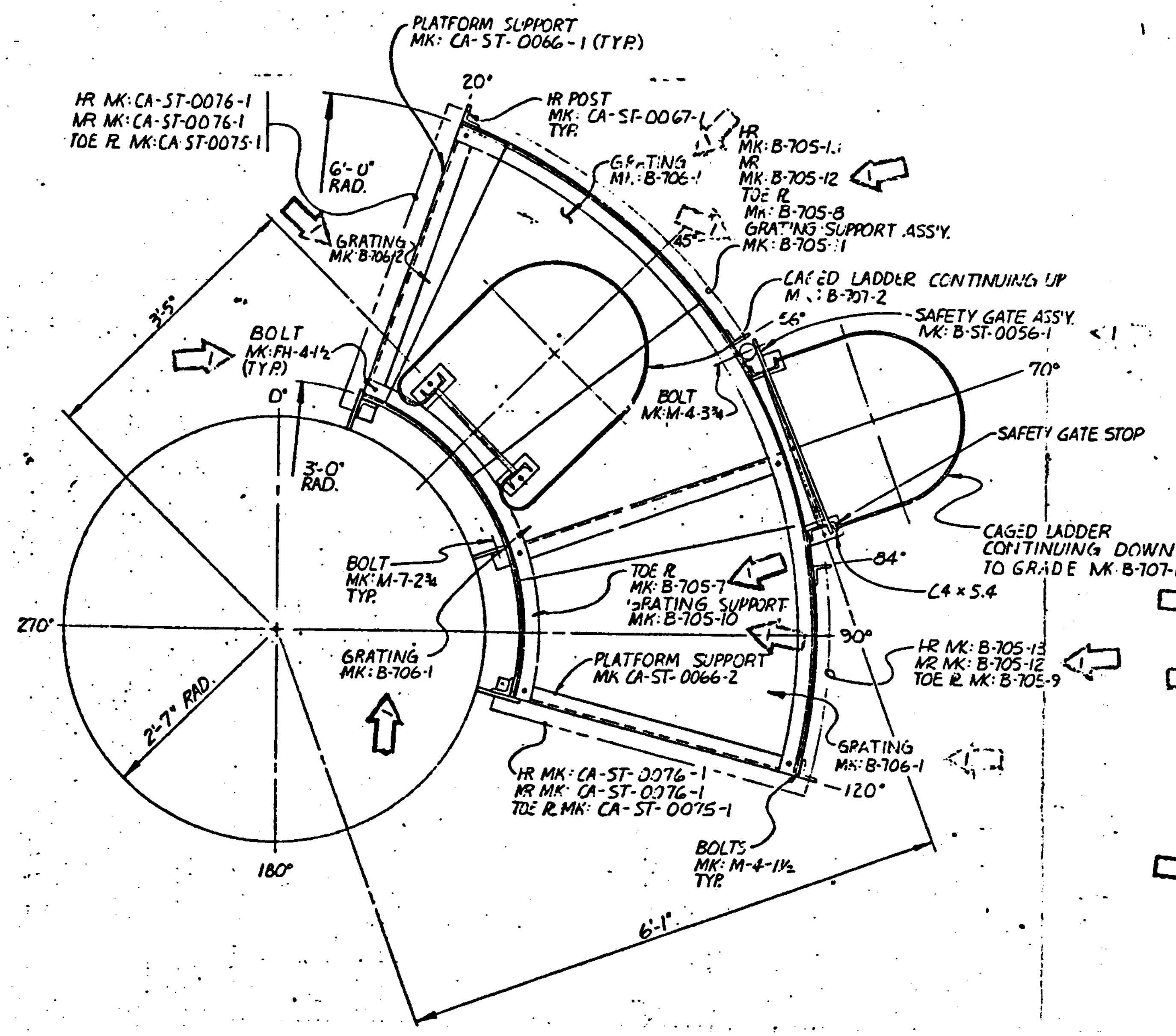
SIDE SECTIONAL ELEVATION
ONE-MK: B-308-1

NO.	REVISION DESCRIPTION	BY	CK.	APP.	DATE
2	REV. PER ECN #2184 & #2185, CHG'D NOZZLE & ELEV.	RJ			3-25-76
1	REVISED PER CUST. REQUEST & ECN #1266	JMT	dh		2-13-76

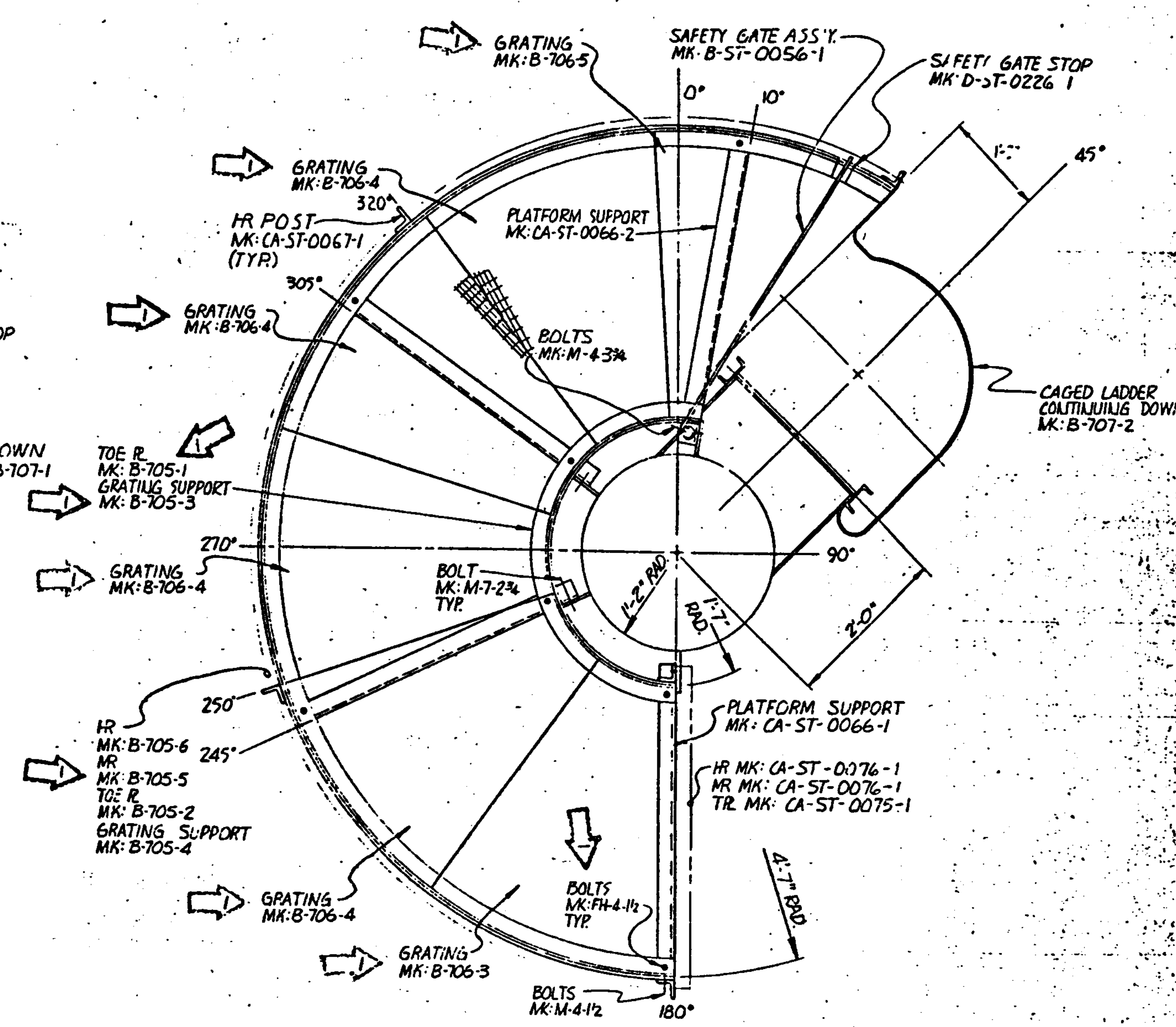
JOHN ZINK COMPANY
TULSA, OKLAHOMA

USER: MONSANTO
JOBSITE: ST. LOUIS, MO.
ABSORBER (UPPER SECTION) DETAIL
S.O. NO. 08181
P.O. NO. 3008-9-0027
FOR: MONSANTO
DR. JW — DATE 12-29-75
CK. RJ — DATE 1-19-76
APP. — DATE 1-2?-76
CERTIFIED — DATE 3-2-76
SCALE
DRAWING NUMBER B-0-81181-308
REV. 2

MONSANTO COMPANY
CORPORATE ENGR. DEPT. Project (CEA) No. 3001
Order No. BXCE-9-0027
Location: St. Louis, Mo.
Equipment No.
Description: Absorber (Upper Section)
Reviewed by
☐ Make Corrections Noted ☐ Issue as Shown



STEP OFF PLATFORM
@ELEV. 120'-0"



SERVICE PLATFORM
@ELEV. 140'-0"

GENERAL NOTES

1. ALL SURFACES TO BE GALVANIZED PER A.S.T.M. SPEC. #A-123
2. ALL PLATFORMS AND LADDERS TO BE IN ACCORDANCE W/OSHA STANDARDS
3. ALL MK NO'S TO BE PRECEDED BY S.O. NO.
4. BOLTS: M-7-2¾ : ⅞"⌀ × 2¾" LG. MACH. BOLT W/ NUT
   M-4-1½ : ½"⌀ × 1½" LG. MACH. BOLT W/ NUT
   M-4-3¾ : ½"⌀ × 3¾" LG. MACH. BOLT W/ NUT
   FH-4-1½ : ½"⌀ × 1½" LG. FLAT HEAD BOLT W/ NUT
5. SADDLE CLIPS W/ SCREWS MK: SC-2-1

REFERENCE DRAWINGS

B-0-81181-203 : GENERAL ARRANGEMENT-SIDE ELEV.
B-ST-0062-3 : PLATFORM STANDARD
B-ST-0064-3 : LADDER STANDARD

MONSANTO COMPANY Order No. 3008-9-0037
CORPORATE ENGR. DEPT. Project (CEA) No. 3008
Location St. Louis, Mo. Area 02
Equipment No. 111 000; 114 000; 165 000
Description [illegible]
Reviewed by [illegible] Date [illegible]
☐ Make Corrections noted ☐ Leave as Shown ☒ First

NO.	REVISION DESCRIPTION	BY	CK.	APP.	DATE
1	ADDED MK. NO'S (ECN #1266)	JWT	dn	DP	2-18-76

JOHN ZINK COMPANY
TULSA OKLAHOMA

PLATFORM DETAILS
(ONE REQ'D.)

USER: MONSANTO		
JOBSITE: ST. LOUIS, MO.		
S.O NO. 08121		
P.O NO 3008-9-0037	FOR: MONSANTO	
DR JW	DATE 1-8-76	CERTIFIED
CK. RN	DATE 1-19-76	DRAWING NUMBER B-0-81121-310 REV 1
APP DT	DATE 1-20-76	DATE 3-23-76 SCALE

Appendix B

Spare Parts Lists

JOHN ZINK PROCESS SYSTEMS
A DIVISION OF THE JOHN ZINK COMPANY
4401 SOUTH PEORIA • TULSA, OKLAHOMA 74105 • PHONE 918/747-1371 • TELEX 49-74-14

June 15, 1976

Monsanto Company
Corporate Engineering Dept.
800 N. Lindbergh Blvd.
St Louis, Missouri 63166

Attention: Jeff Waldbeser

Reference: Your P.O.: 3008-9-0037
John Zink S.O. 081181

Gentlemen:

Attached are copies of recommended spare parts for the equipment supplied for the above referenced order.

If there are any questions, please do not hesitate to contact us.

Very truly yours,

JOHN ZINK COMPANY

Dick Pipkin
Project Engineer

DP:pas
Enclosures

JOHN ZINK PROCESS SYSTEMS
A DIVISION OF THE JOHN ZINK COMPANY
4401 SOUTH PEORIA • TULSA, OKLAHOMA 74105 • PHONE 918-747-1371 • TELEX 49-74-14

RECOMMENDED SPARE PARTS

EQUIPMENT Quench Gun Assembly DATE June 14, 1976

CUSTOMER Monsanto

CUSTOMER P.O. NO. 3008-9-0037

JOHN ZINK S.O. NO. 081181

JOHN ZINK DWG. NO. B-0-81181-606

ITEM	QTY.	DESCRIPTION	PART NO.	UNIT PRICE	DEL. WKS.
	2	Quench Gun Assembly	B-0-81181-606	*	*
	4	Tip (Spraying Systems)	1/2 GG-32 Titanium	*	*
		* Price and delivery at inquiry.			

NOTES:

1. PRICES SUBJECT TO CHANGE WITHOUT NOTICE
2. MINIMUM INVOICE $100
3. SHIPMENT: F.O.B. POINTS OF MANUFACTURE
4. TERMS: NET 30 DAYS
5. WHEN ORDERING, REFER TO DRAWING AND S.O. NUMBERS ABOVE.

JOHN ZINK PROCESS SYSTEMS
A DIVISION OF THE JOHN ZINK COMPANY
4401 SOUTH PEORIA • TULSA, OKLAHOMA 74105 • PHONE 918/747-1371 • TELEX 49-74-14

RECOMMENDED SPARE PARTS

EQUIPMENT DH Waste Gun - Gun No. 4 DATE June 14, 1976

CUSTOMER Monsanto

CUSTOMER P.O. NO. 3008-9-0037

JOHN ZINK S.O. NO. 081181

JOHN ZINK DWG. NO. A-0-81181-607

ITEM	QTY.	DESCRIPTION	PART NO.	UNIT PRICE	DEL. WKS.
	1	DH - Waste Gun Insert	A-0-81181-607	805.00	6
	1	Atomizer (Hast C)	DH-1	280.00	6
	1	DH - Waste Tip (Hast C)	DH tip	245.00	6
	1	Sleve (Hast C)	DH-SLV	220.00	6
	1	Waste Body Receiver (Hast C)	RZ-56-1	190.00	6
	1	Waste Body (Hast C)	RZ-56-2	170.00	6
	10	Gasket	RZ-56-6T	1.25	6
	10	Gasket O Ring	2-1/8 OD x 1-3/4 ID	3.50	6

NOTES:

1. PRICES SUBJECT TO CHANGE WITHOUT NOTICE
2. MINIMUM INVOICE $100
3. SHIPMENT: F.O.B. POINTS OF MANUFACTURE
4. TERMS: NET 30 DAYS
5. WHEN ORDERING, REFER TO DRAWING AND S.O. NUMBERS ABOVE.

JOHN ZINK PROCESS SYSTEMS
A DIVISION OF THE JOHN ZINK COMPANY
4401 SOUTH PEORIA • TULSA, OKLAHOMA 74105 • PHONE 913/747-1371 • TELEX 49-74-14

RECOMMENDED SPARE PARTS

EQUIPMENT HI 24 Burner Assembly DATE June 15, 1976

CUSTOMER Monsanto

CUSTOMER P.O. NO. 3008-9-0037

JOHN ZINK S.O. NO. 081181

JOHN ZINK DWG. NO. B-0-81181-601

ITEM	QTY.	DESCRIPTION	PART NO.	UNIT PRICE	DEL. WKS.
	1	Sight Glass	4" OD x 3/4" THK	35.00	6
	20 ft.	Garlock Rope	1/4"		

NOTES:

1. PRICES SUBJECT TO CHANGE WITHOUT NOTICE
2. MINIMUM INVOICE $100
3. SHIPMENT: F.O.B. POINTS OF MANUFACTURE
4. TERMS: NET 30 DAYS
5. WHEN ORDERING, REFER TO DRAWING AND S.O. NUMBERS ABOVE.

JOHN ZINK PROCESS SYSTEMS
A DIVISION OF THE JOHN ZINK COMPANY
4401 SOUTH PEORIA • TULSA, OKLAHOMA 74105 • PHONE 918/747-1371 • TELEX 49-74-14

RECOMMENDED SPARE PARTS

EQUIPMENT Thermal Oxidizer DATE June 14, 1976

CUSTOMER Monsanto

CUSTOMER P. O. NO. 3008-9-0037

JOHN ZINK S.O. NO. 081181

JOHN ZINK DWG. NO. D-ST-0238-3 (Sight Port)

ITEM	QTY.	DESCRIPTION	PART NO.	UNIT PRICE	DEL. WKS.
		Sight Glass - 6" OD x 3/4" THK Pyrex		35.00	6

NOTES:

1. PRICES SUBJECT TO CHANGE WITHOUT NOTICE
2. MINIMUM INVOICE $100
3. SHIPMENT: F.O.B. POINTS OF MANUFACTURE
4. TERMS: NET 30 DAYS
5. WHEN ORDERING, REFER TO DRAWING AND S.O. NUMBERS ABOVE.

JOHN ZINK PROCESS SYSTEMS
A DIVISION OF THE JOHN ZINK COMPANY
4401 SOUTH PEORIA • TULSA, OKLAHOMA 74105 • PHONE 918/747-1371 • TELEX 49-74-14

RECOMMENDED SPARE PARTS

EQUIPMENT HI - 24 Burner Plenum DATE 6-14-76

CUSTOMER Monsanto

CUSTOMER P.O. NO. 3008-9-0037

JOHN ZINK S.O. NO. 081181

JOHN ZINK DWG. NO. B-0-81181-305

ITEM	QTY.	DESCRIPTION	PART NO.	UNIT PRICE	DEL. WKS.
	1	HI-24 Burner Tile		*	*
		Per: B-0-81181-305 with Hast. C Tile Case			
		* Pricing and delivery at inquiry.			

NOTES:

1. PRICES SUBJECT TO CHANGE WITHOUT NOTICE
2. MINIMUM INVOICE $100
3. SHIPMENT: F.O.B. POINTS OF MANUFACTURE
4. TERMS: NET 30 DAYS
5. WHEN ORDERING, REFER TO DRAWING AND S.O. NUMBERS ABOVE.

JOHN ZINK PROCESS SYSTEMS
A DIVISION OF THE JOHN ZINK COMPANY
4401 SOUTH PEORIA • TULSA, OKLAHOMA 74105 • PHONE 918/747-1371 • TELEX 49-74-14

RECOMMENDED SPARE PARTS

EQUIPMENT EA - Oil Gun Insert - Gun No. 1 DATE 6-14-76

CUSTOMER Monsanto

CUSTOMER P.O. NO. 3008-9-0037

JOHN ZINK S.O. NO. 081181

JOHN ZINK DWG. NO. A-0-81181-605

ITEM	QTY.	DESCRIPTION	PART NO.	UNIT PRICE	DEL. WKS.
	1	EA Oil Gun Insert	A-0-81181-605	775.00	6
	1	EA Tip (Hast C)	EA-765	250.00	6
	1	Sleve (Hast C)	225	220.00	6
	1	Atomizer (303 S.S.)	EA-7	220.00	6
	1	Oil Body Receiver	RZ-56-1	70.00	6
	1	Oil Body	TZ-56-2	55.00	6
	10	Gasket	RZ-56-6	.40	6
	10	Gasket O Rings	2-1/8 OD x 1-3/4 ID	3.50	

NOTES:

1. PRICES SUBJECT TO CHANGE WITHOUT NOTICE
2. MINIMUM INVOICE $100
3. SHIPMENT: F.O.B. POINTS OF MANUFACTURE
4. TERMS: NET 30 DAYS
5. WHEN ORDERING, REFER TO DRAWING AND S.O. NUMBERS ABOVE.

JOHN ZINK PROCESS SYSTEMS
A DIVISION OF THE JOHN ZINK COMPANY
4401 SOUTH PEORIA • TULSA, OKLAHOMA 74105 • PHONE 913/7[illegible]-13[illegible] • TELEX 49-74-14

RECOMMENDED SPARE PARTS

EQUIPMENT DH Waste Gun Assembly - Gun #3 DATE 6-14-76

CUSTOMER Monsanto

CUSTOMER P.O. NO. 3008-9-0037

JOHN ZINK S.O. NO. 081181

JOHN ZINK DWG. NO. A-0-81181-604

ITEM	QTY.	DESCRIPTION	PART NO.	UNIT PRICE	DEL. WKS.
	1	DH Waste Gun Assembly (Hast C)	A-0-81181-604	805.00	6
	1	Atomizer (Hast C)	DH-4	280.00	6
	1	Sleve (Hast C)	DH-SLV	220.00	6
	1	Waste Body Receiver (Hast C)	RZ-56-1	190.00	6
	1	Waste Body (Hast C)	RZ-56-2	170.00	6
	10	Gasket	RZ-56-6T	1.25	6
	10	Gasket O Ring	2-1/8 OD x 1-3/4 ID	3.50	6
	1	DH Waste Tip	DH-TIP	245.00	6

NOTES:

1. PRICES SUBJECT TO CHANGE WITHOUT NOTICE
2. MINIMUM INVOICE $100
3. SHIPMENT: F.O.B. POINTS OF MANUFACTURE
4. TERMS: NET 30 DAYS
5. WHEN ORDERING, REFER TO DRAWING AND S.O. NUMBERS ABOVE.

JOHN ZINK PROCESS SYSTEMS
A DIVISION OF THE JOHN ZINK COMPANY
4401 SOUTH PEORIA • TULSA, OKLAHOMA 74105 • PHONE 918/747-1371 • TELEX 49-74-14

RECOMMENDED SPARE PARTS

EQUIPMENT EA Waste Gun Insert - Gun No. 2 DATE 6-14-76

CUSTOMER Monsanto

CUSTOMER P.O. NO. 3008-9-0037

JOHN ZINK S.O. NO. 081181

JOHN ZINK DWG. NO. A-0-81181-603

ITEM	QTY.	DESCRIPTION	PART NO.	UNIT PRICE	DEL. WKS.
	1	Spare "EA" Waste Gun	A-0-81181-603	810.00	6
	1	EA - Waste Tip (Hast C)	EA-765	250.00	6
	1	Sleve (Hast C)	225	220.00	6
	1	Atomizer (Hast C)	EA-7	220.00	6
	1	Waste Body Receiver (Hast C)	RZ-56-1	190.00	6
	1	Waste Body (Hast C)	RZ-56-2	170.00	6
	10	Gasket	RZ-56-6T	1.25	6
	10	Gasket O Ring	2-1/8" O.D. x 1-3/4 I.D.	3.50	6

NOTES:

1. PRICES SUBJECT TO CHANGE WITHOUT NOTICE
2. MINIMUM INVOICE $100
3. SHIPMENT: F.O.B. POINTS OF MANUFACTURE
4. TERMS: NET 30 DAYS
5. WHEN ORDERING, REFER TO DRAWING AND S.O. NUMBERS ABOVE.

JOHN ZINK PROCESS SYSTEMS
A DIVISION OF THE JOHN ZINK COMPANY
4401 SOUTH PEORIA • TULSA, OKLAHOMA 74105 • PHONE 918/747-1371 • TELEX 49-74-14

RECOMMENDED SPARE PARTS

EQUIPMENT Tempering Gun DATE 6-14-76

CUSTOMER Monsanto

CUSTOMER P.O. NO. 3008-9-0037

JOHN ZINK S.O. NO. 081181

JOHN ZINK DWG. NO. B-0-81181-602

ITEM	QTY.	DESCRIPTION	PART NO.	UNIT PRICE	DEL. WKS.
	1	EA Gun Insert	B-0-81181-602	400.00	6
	1	EA Tip	EA-1064	100.00	6
	1	EA Atomizer	EA-10	100.00	6
	1	Sleve	225	35.00	6
	1	Oil Body Receiver	RZ-56-1	70.00	6
	1	Oil Body	RZ-56-2	55.00	6
	10	Gasket	Z-56-6	.40	6
	10	Gasket O Ring	2-1/8 OD x 1-3/4 ID	3.50	6

NOTES:

1. PRICES SUBJECT TO CHANGE WITHOUT NOTICE
2. MINIMUM INVOICE $100
3. SHIPMENT: F.O.B. POINTS OF MANUFACTURE
4. TERMS: NET 30 DAYS
5. WHEN ORDERING, REFER TO DRAWING AND S.O. NUMBERS ABOVE.

JOHN ZINK PROCESS SYSTEMS
A DIVISION OF THE JOHN ZINK COMPANY
4401 SOUTH PEORIA • TULSA, OKLAHOMA 74105 • PHONE 918/747-1371 • TELEX 49-74-14

RECOMMENDED SPARE PARTS

EQUIPMENT HI Pilot DATE 6-14-76

CUSTOMER Monsanto

CUSTOMER P.O. NO. 3008-9-0037

JOHN ZINK S.O. NO. 081181

JOHN ZINK DWG. NO. B-0-81181-608

ITEM	QTY.	DESCRIPTION	PART NO.	UNIT PRICE	DEL. WKS.
	1	Pilot Tip (Hast C)	ST-6-1-1/2	**	*
	1	Pilot Mixer Assembly (Hast C)	ST-6-1-1/2 AHR	*	*
	2	Ignition Rod (Hast C)	CA-ST-0100	*	*
	1	Connecting Coupling (Hast C)	CA-PAT-846	*	*
	1	Ignition Rod Extension (Hast C)	3/16"	*	*
	1	Spark Plug	JZ-WCC-12-1K	10.00	6
	2	Insulator	JZ-E-27782	5.00	6
	1	Corrosion Seal (Teflon)	A-0-81181-713	*	*
		* Price and delivery at Inquiry.			

NOTES:

1. PRICES SUBJECT TO CHANGE WITHOUT NOTICE
2. MINIMUM INVOICE $100
3. SHIPMENT: F.O.B. POINTS OF MANUFACTURE
4. TERMS: NET 30 DAYS
5. WHEN ORDERING, REFER TO DRAWING AND S.O. NUMBERS ABOVE.

Appendix C

CAC Incinerator Procedures

WASTE OXIDIZER

START-UP PROCEDURE

1. Start combustion air blower, 02-169.000

   A. Incinerator blower "auto-man." switch to manual.
   B. Start blower.

2. Turn fuel selector switch to fuel desired, fuel gas or fuel oil.

3. Check to see that main block valves for fuel selected are in full open position.

4. Turn "low oxidizer temp" switch to "by-pass" position.

5. Turn "residue to receiver" switch to "by-pass" position.

6. Turn "Azo residue flow to oxidizer" switch to "close" position.

7. Turn "organic waste flow to oxidizer" switch to "close" position.

8. Turn "HAC bottom flow to oxidizer" switch to "close" position.

9. Set TIC 114-1 on manual and move set point to lowest temperature (Ambient). Observe TJI 114-1 and TJI 114-7.

0. Open hand valve from nitrogen header to flame safeguard system.

11. If fuel gas is selected;

   A. Start water booster pump, 02-178 or 02-178.010. Check the water level in absorber and quench pot. If the water level is above the "low quench pot/absorber water level" start the acid return pump 2-135 or 2-135.01
   B. Start waste oxidizer

      (1) "Blue" purging ind. light on

      (2) 2 min. later purging light off

      (3) "Amber" purging complete light on

      (4) "Red" pilot blocking valves open ind. light on

      (5) Observe ~~light off~~ flame on, 15 sec. later

12. If fuel oil is selected;

   A. Atomizing steam for fuel oil gun should be turned on and regulated to 100 psig. Both sides of the oil gun should be

blown down with steam by opening the crossover valve between the oil and steam lines.

B. Close the crossover valve.

C. Start oil pump, 02-226.10 or 02-266.51

D. Start water booster pump, 02-178 or 02-178.010. Check the water level in absorber and quench pot. If water level is above the "low quench pot/absorber water level", start the acid return pump 2-135 or 2-135.01

E. Start waste oxidizer

(1) "Blue" purging ind. light on

(2) 2 min. later purging light off

(3) "Amber" purging complete light on.

(4) "Red" pilot blocking valves open ind. light on.

(5) "Red" fuel oil on, 15 sec. later

(6) Observe light off.

Note: Before attempting to raise the temperature any higher the Initial Refractory Curing Instructions should be read and understood. The Time U.S. Temperature Firing Schedule must be followed for refractory curing.

13. To increase the system temperature, switch TIC 114-1 to automatic mode and increase set point to desired temperature.

Note: TIC 114-1 can indicate 800°C to 1400°C only. So use TJI 114-1 and 114-7 to observe 0°C to 800°C

14. When Thermal Oxidizer refractory has been cured completely and system is at design operating temperature, tempering water gun should be placed in the proper receiver and atomizing steam turned on to the gun. Again both sides of the gun should be blown down with steam by opening the steam line crossover valve for about 1 minute. Close steam line crossover valve.

15. Check to insure that tempering water atomizing steam is on and pressure regulated to 25 psig differential pressure above water. (about 100 psig)

16. Open block firing valve on tempering water line.

~~17. Check the water level in absorber and quench pot.~~

18. ~~If water level is above the "low quench pot/absorber water level", start the acid return pump 2-135 or 2-135.01.~~

19. At this point each liquid stream may be introduced one at a time letting the temperature stabilize sufficiently between stream introductions. The method for introducing Monsanto's waste streams #1, 2 and 3 are the same methods used to introduce the fuel oil and tempering water.

20. The waste gas streams #4 and 5 may now be introduced one at a time again letting the temperature stabilize between streams.

21. Adjust the valve on the overflow line to clean acid sewer to stabilize water level in the quench pot and absorber.

Since the composition of some of the waste streams are exothermic during normal operation the main burner will slow to minimize firing rates and the tempering water guns will come on to maintain the correct operating temperature in the Thermal Oxidizer.

At this point providing all the instrumentation is set properly and nothing is abnormal as far as the design criteria is concerned, the system should run on complete automatic mode.

TOTAL SHUTDOWN LIMITS

The Thermal Oxidizer is automatically shutdown for the following reasons:

1. Flame Failure (pilot and main)
2. Low Gas Pressure/Low Fuel Oil Pressure
3. Thermal Oxidizer High Temperature
4. Thermocouple Failure
5. Electrical Power Failure
6. Scanner or Scanner Relay Failure
7. Contactor Low Water Flow (recycle water to quench pot)
8. Absorber water low flow
9. Water weir low flow (in quench pot)
10. Tempering water (oxidizer water) Low Flow
11. High Quench Pot Temperature

With automatic shutdown due to a malfunction, the unit is subjected to the same problems as emergency shutdown. Water to the quench section must be checked for proper flow to prevent thermal damage to the FRP equipment.

If the unit must be taken off stream for several hours, every attempt must be made to cool the unit according to the normal cooling schedule to prevent unnecessary damage to the refractory and other equipment.

NORMAL START-UP CHECK LIST

After the initial start-up check list has been satisfied, the following items given in the Initial Start-Up Instructions section should be re-checked before each normal start-up is initiated.

7. Manual Valves - All manual valves should be in the closed position.

8. Pressures - Pressures should be regulated as given in Initial Start-Up Check List.

9. Wastes - Liquid and gaseous wastes are blocked from the system.

10. Quench Water - Quench water is regulated as given in Initial Start-Up Check List.

11. Absorption Column Recycle and Fresh Water Make Up - These items are regulated as given in Initial Start-Up Check List.

12. Quench Pot - [redacted] - Level is regulated to be even with the top of the serrations in the quench pot downcomer.

13. Waste Gun - Waste gun should be checked as given in Initial Start-Up Check List.

INITIAL REFRACTORY CURING INSTRUCTIONS

The Thermal Oxidizer should be heated up according to the following Time-Temperature Schedule, after a minimum of one day of Air Curing.

Time-Hours	OXIDIZER TEMPERATURES	
0-1	Raise to 300°F	(149°C)
1-7	Hold at 300°F	(149°C)
7-8	Raise to 350°F	(177°C)
8-9	Raise to 400°F	(204°C)
9-10	Raise to 450°F	(232°C)
10-11	Raise to 500°F	(260°C)
11-12	Raise to 550°F	(288°C)
12-13	Raise to 600°F	(316°C)
13-14	Raise to 650°F	(343°C)
14-15	Raise to 700°F	(371°C)
15-16	Raise to 750°F	(399°C)
16-17	Raise to 800°F	(427°C)
17-18	Raise to 850°F	(454°C)
18-19	Raise to 900°F	(482°C)
19-20	Raise to 950°F	(510°C)
20-21	Raise to 1000°F	(538°C)
21-31	Hold at 1000°F	(538°C)
31-32	Raise to 1200°F	(649°C)
32-34	Raise to 1400°F	(760°C)
34-36	Raise to 1600°F	(871°C)
36-38	Raise to 1800°F	(982°C)
38-40	Raise to 2000°F	(1093°C)
40-42	Hold at 2000°F	(1093°C)

NOTE: Once the curing is started, the Thermal Oxidizer should not be turned off until curing is complete.

If the unit has been shutdown for any length of time, the following schedule should be followed to prevent excessive damage to the refractory.

(1) Extended shutdown one month or longer: Complete cure in compliance with initial curing cycle.

(2) One week to one month shutdown: Heat system with pilot only for 24 hours. Raise temperature per Cold Start-Up procedure that follows.

(3) Maximum shutdown one week: Heat system for one hour with pilot. Raise temperature per Cold Start-Up procedure that follows.

NORMAL COLD START-UP

AFTER INITIAL REFRACTORY CURE-OUT

TIME-HOURS	OXIDIZER TEMPERATURE
0-1	Raise to 500°F
1-4	Hold at 500°F
4-5	Raise to 600°F
5-6	Raise to 800°F
6-7	Raise to 1000°F
7-8	Raise to 1200°F
8-9	Raise to 1400°F
9-10	Raise to 1600°F
10-11	Raise to 1800°F
11-12	Raise to 2000°F

NORMAL HOT START-UP

AFTER TEMPORARY SHUTDOWN

Over a period of 30 minutes, gradually increase the temperature to 1000°F. Hold this temperature for one hour. Gradually increase the temperature to 2000°F.

JOHN ZINK PROCESS SYSTEMS

TULSA OKLAHOMA

NORMAL SHUTDOWN

1a OPEN THE VALVE ON THE LIQUID WASTE RECYCLE LINE.

1b Stop liquid waste flow to the system by closing manual feed valve.

2. Open manual steam valve supplying steam to liquid waste gun and purge waste gun of residual liquid waste.

3. Close manual steam purge and atomizing valves.

4. Open waste gun air seal manual air valve supplying air to seal.

5. Loosen clevis and remove liquid waste gun.

6. Insert receiver plug and secure clevis. Close air seal valve.

7. Shut off gaseous wastes.

8. Decrease operating temperature 200°F every hour by changing set point of temperature indicator controller (6) until 200°F is reached. If 200°F cannot be reached with burner on, close manual fuel gas valve and adjust blowers to maintain proper temperature schedule.

9. Close fuel gas manual valves.

10. After flame shutdown turn off blowers. Leave water circulating to avoid damage to FRP equipment.

11. Turn off water supply to water weir, contactor tube, and [redacted] absorption column after temperature has reached ambient.

12. Turn the "Power On-Off" switch [redacted] to the "Off" position.

EMERGENCY SHUTDOWN

1. Switch "Power On-Off" switch [redacted] to the "Off" position.
2. Turn all blowers off.
3. Check to be sure water is flowing to the water weir, contactor tube, and vent stack (absorption column).
4. Pull liquid waste gun and secure receiver plug.

NOTE: This form of shutdown can result in damage to the Thermal Oxidizer, Refractory, and other related equipment and should be avoided.

TROUBLESHOOTING GUIDE

INCINERATOR (WASTE OXIDIZER)

SYMPTOM	PROBLEM	CAUSE	SOLUTION
Steam pressure to incinerator alarm activates	Low steam pressure	Atomizing steam to quench water gun is low, causing poor water atomization	Check manual valves and field gauge for problem area
Incinerator quench water alarm activates	Low water pressure - below 30 psig	Quench water flow to incinerator has been stopped by temperature controller	Correct and watch incinerator temperature
Main/pilot gas pressure alarm activates	High gas pressure. Gas pressure is above 16 psig	Malfunction in gas supply controls	Check controls and correct problem
	Low gas pressure. Gas pressure is below 3 psig	Malfunction in gas supply controls	Check controls and correct problem
		Low gas pressure may cause flameout. This shuts down the incinerator by interlocks	Restart incinerator checking for low gas pressure
Incinerator feed tank flow alarm activates	High/low feed tank flow above/below 1900/500 lb/hr	Circulating flow from feed tank has stopped	Check feed tank circulating pumps

TROUBLESHOOTING GUIDE

SYMPTOM	PROBLEM	CAUSE	SOLUTION
Fuel oil pressure alarm activates	Low fuel oil pressure - less than 30 psig	Oil storage tank low level	Check storage tank level; if low order fuel oil
		Oil feed pumps have shut down	Restart pumps, determine why they shut down. Restart incinerator.
Steam pressure to feed gun alarm activates	Steam pressure is below 20 psig. Low atomizing steam pressure to feed guns causes poor waste burning. Interlock shuts off feed to that gun.	Malfunction in steam supply system	Check steam pressure to guns. Correct malfunction and restart feed
Low temperature in incinerator quench chamber. Temp. below 875°C	Temperature in incinerator is too low to decompose waste feed streams. Interlock shuts feed streams off, stops water flow to quench chamber and diverts gaseous wastes to vent stack	Waste feed, steam pressure, fuel feed, or combustion air flow problems	Re-establish temperature and restart waste feeds

TROUBLESHOOTING GUIDE

SYMPTOM	PROBLEM	CAUSE	SOLUTION
High temperature in incinerator quench chamber above 1100°C	High temperature in incinerator may cause equipment damage. Interlock stops waste feeds, gas/oil, steam quench water and diverts gaseous wastes to vent stack	Malfunction in quench water feed	Check water feed, feed pumps and supply. Correct problem and restart unit.
High temperature in quench pot, above 135°C	High temperature may damage the equipment. Interlock shuts down incinerator	Malfunction in quencher water flows	Same as above solution
Low water flow at incinerator/ scrubber. Flows below 30/5/5 gpm	Low water flow results in high incinerator temperatures and equipment damage	Malfunction in water supply, pump failure, regulator failure, etc.	Check pump operation and HV setting. Re-establish flows and restart unit.
Low air combustion flow, below 750 cfm	Poor waste burning in incinerator. Excessive O_2 in incinerator	Malfunction in air blower system	Check excess O_2 in incinerator. Correct problem in blower system

TROUBLESHOOTING GUIDE

SYMPTOM	PROBLEM	CAUSE	SOLUTION
Fume blower alarm activates	Fume blower has stopped	Fume blower malfunctions	Check blower operation, switch to spare if necessary
Incinerator scrubber pump alarm activates	Scrubber pump has shut down	Scrubber pump malfunctions	Check pump operation, restart pump or switch to spare if necessary.
Incinerator water booster pump alarm activates	Water booster pump has shut down	Malfunction with water booster pump	Check pump operation, restart or switch to spare pump
HIGH/LOW LEVEL CONDENSATE TANK			
High/low level alarm activates on condensate tank	High level causes back-pressure in condensate drain	Condensate pumps have shut down	Check field level gauge, check pump operation, open drain to sewer if necessary
	Low level indicates an empty tank	Condensate pump has pumped tank empty or drain valve is open	Check pumps and drain valve. Correct if necessary.

Appendix D

Nonstandard Sampling and Analytical Methods

Monsanto Agricultural Research

Analytical Report

Analyte :	1,2-DICHLOROETHANE (DCE)	Method Number :	130
Matrix :	CAC WASTE	Validation Date:	1-15-85
Procedure:	ADD TOLUENE (IS), DILUTE WITH CH2CL2, ANALYSE BY CAPILLARY GC/FID	Range:	0.1% - 7.1%
		Author(s):	D L KLEYER T E NEUMANN

1.0 PRINCIPLE OF METHOD

1.1

Described in this method is a capillary gas chromatographic procedure for the determination of 1,2-dichloroethane (DCE, CP 6100) in CAC waste. CAC waste is a combination of still bottoms and light ends from the distillation of chloroacetyl chloride (CAC).

1.2

The internal standard toluene is added to a volumetric flask containing an accurately weighed sample of CAC waste and diluted with dichloromethane to volume.

1.3

The dichloromethane solution is injected into a capillary gas chromatograph equipped with a 60 m X 0.25 mm ID DB-5 fused silica open tubular column and a flame ionization detector (FID).

1.4

Quantitation is based on the ratio of peak areas, analyte:toluene, as measured by an electronic integrator.

2.0 SAFETY

2.1

The analyte and solvent used in this method should be used only with proper precautions and adequate ventilation. Contact with CAC samples should be minimized due to their corrosive and odoriferous nature.

3.0 RANGE AND SENSITIVITY

3.1 Range

The laboratory validation of the method covered the range 0.1 - 7.1 %. The applicable range can easily be extended through a change in the sample size. The current validation is based on a 1300 mg sample. The above range adequately covers the levels observed for DCE in previous samples with only one exception.

3.2 Sensitivity

The lower limit of validation for DCE is 0.1% based on a 1300 mg sample. The lower limit can easily be extended by increasing the sample size.

4.0 INTERFERENCES

4.1

This method was developed to provide adequate resolution between the various peaks of interest which are found in typical CAC waste. It was found that the FSOT capillary column used needed to be conditioned by baking the column at 300 degrees under a flow of helium carrier gas overnight. Conditioning was needed upon installation of the column and approximately once every week. The capillary glass injector insert should be changed at these times. Failure to do this produced large, irreproducible peaks superimposed on the expected chromatogram. No other interferences were found during the analysis of any CAC waste sample.

5.0 PRECISION AND ACCURACY

5.1 Precision

Coefficients of variation (CV) were calculated using Valmet [1] data analyses according to NIOSH-type Statistical testing of the validation results. The CV's ranged from 0.4 to 3.2%. Bartlett's Test for Equality of Variance was not met. Attachment 1 lists the raw data used in the Valmet analyses. The levels reported are in weight percent of CAC waste.

5.2 Accuracy

Validations were run by spiking a standard solution of the analyte into a sample of the waste which was fortuitously low in the level of analyte present. The slope and intercept for the equation of prediction were 1.00 and 0.01 respectively with a correlation coefficient of 1.0000. The average recovery was 95.8%. The data show good correlation between the laboratory validation analyses compared with their known spiked levels.

6.0 ADVANTAGES AND DISADVANTAGES

6.1 Advantages

This method is very sensitive and reproducible. Little sample preparation is necessary. Sample analysis is accomplished rapidly by an instrumental method. The equipment, solvents and internal standard are commercially available. Interferences were nonexistant in the samples analyzed. The use of capillary gas chromatographic technology allows the baseline resolution of a complex mixture of components.

6.2 Disadvantages

Capillary gas chromatography requires more specialized equipment than does packed column gas chromatography. Odoriferous samples must be handled.

7.0 APPARATUS

7.1

Gas Chromatograph. A Varian 6000 gas chromatograph (or equivalent) equipped with a split injector (for capillary columns), a flame ionization detector (FID), and a Varian autosampler.

7.2

Gas Chromatographic Column. J and W Sientific, 3871 Security Park Drive, Rancho Cordova, CA 95670, 60m X 0.25 mm ID DB-5 (catalogue number 122-5062).

7.3

Recording Integrator. An in-house computerized recording integration system (Monsanto Chromatographic Data System) was used. A Hewlett Packard 3390 recording integrator was used as backup and for real time monitoring.

7.4

Sample Vials. Varian Associates, Instrument Group Service Center, 220 Humboldt Court, Sunnyvale, CA 94086, 2mL septum vials with open-top cap and teflon-silicone septum (catalogue number 96-000099-00).

7.5

Assorted Laboratory Glassware. Pipettes, volumetric flasks, syringes, etc.

8.0 REAGENTS

8.1

Solvent. Dichloromethane; Burdick and Jackson distilled in glass grade, Burdick and Jackson Laboratories Inc., 1953 South Harvey Street, Muskegon, MI 49442 (catalogue number 300).

8.2

Internal Standard. Toluene; Burdick and Jackson distilled in glass grade, Burdick and Jackson Laboratories Inc., 1953 South Harvey Street, Muskegon, MI 49442 (catalogue number 347).

8.3

Standard Materials. 1,2-Dichloroethane; Fisher Scientific, 1241 Ambassador Blvd., P.O. Box 14989, St. Louis, MO 63178 (catalogue number E-175).

8.4

Preparation of Internal Standard Solution. Into a 25 mL volumetric flask pipet in 2 mL of toluene Dilute to the mark with dichloromethane. This solution contains 69360 mg/L toluene internal standard.

8.5

Preparation of Analyte Stock Solution. Into a 25 mL volumetric flask pipet 5 mL of 1,2-dichloroethane (DCE). Dilute to the mark with dichloroethane. This solution contains 251200 mg/L DCE.

9.0 CALIBRATION AND STANDARDIZATION

The composition of standards used for instrument calibration is as follows:

Solution A. Into a 25 ml volumetric flask are transfered 0.4 mL of analyte stock solution and 0.05 mL of internal standard stock solution. Dilute to the mark with dichloromethane. The concentration of DCE in this standard is 4019.2 mg/L.

Solution B. Into a 25 mL volumetric flask are transfered 0.2 mL of analyte stock solution and 0.05 mL of internal standard stock solution. Dilute to the mark with dichloromethane. The concentration of DCE in this standard is 2009.6 mg/L,

Solution C. Into a 25 mL volumetric flask are transfered 0.05 ml of analyte stock solution and 0.05 mL of internal standard stock solution. Dilute to the mark with dichloromethane. The concentration of DCE in this standard is 502.4 mg/L.

Solution D. Into a 25 mL volumetric flask are transfered 0.02 mL of analyte stock solution and 0.05 mL of internal standard stock solution. Dilute to the mark with dichloromethane. The concentration of DCE in this standard is 200.96 mg/L.

Solution E. Into a 25 mL volumetric flask are transfered 0.005 mL of analyte stock solution and 0.05 mL of internal standard stock solution. Dilute to the mark with dichloromethane. The concentration of DCE in this standard is 50.24 mg/L.

Instrument Calibration

Make three 1 microliter injections of Solution C (prepared above) to assure that the system is conditioned and that detector response is consistent.

Analyze aliquots of Solutions A - E on the gas chromatograph to prepare a standard response curve of detector response (peak area)

versus solution concentration in mg/L. A least-squares treatment of data is recommended. Response curves generated in this manner must have correlation coefficients of greater than 0.99 before proceeding. A typical calibration chromatogram is shown in Attachment 2. Typical retention times for DCE and toluene are 428 and 652 sec respectively.

10.0 PROCEDURE

10.1 Cleaning Of Equipment

10.1.1
It is necessary to clean all glassware carefully and scrupulously before use to avoid possible sources of contamination and cross-contamination. Use of standard good laboratory practices for cleaning glassware is encouraged.

10.2 Collection And Shipping Of Samples

10.2.1
Samples should be collected in glass containers with teflon or polypropylene-lined lids to avoid contamination. Sample containers must be scrupulously clean.

10.2.2
After the sample is collected mark the container with the appropriate information for future identification.

10.2.3
Exposure to extreme heat and direct sunlight for prolonged periods (several days) must be avoided. This can cause loss of analyte.

10.3 Sample Preparation

10.3.1
Accurately weigh approximately 1300 mg into a 25 mL volumetric flask. Add 0.05 mL of internal standard solution. Dilute to the mark with dichloromethane. Mix the contents of the flask thoroughly. The sample is now ready for analysis.

10.4 Analysis Of Prepared Sample

10.4.1
To assure a high degree of stability in the chromatographic system, make several injections of a single standard. Follow the procedure in Section 9 for the preparation of a standard response curve.

10.4.2
Using the conditions described in Section 10.4.5 make at least 2 injections of the prepared sample into the gas chromatograph. When using an autosampler it is useful to position wash vials between standards and samples to avoid carry-over. The use of wash vials after samples is necessary to prevent autosampler corrosion. An example chromatogram from the analysis of a sample of CAC waste is shown in Attachment 3. DCE was assigned on the basis of retention time and GC/MS spectral information obtained under similar conditions.

10.4.3
Results are calculated from peak areas of DCE and toluene internal standard as described in Section 11 (calculations).

10.4.4
Frequent standards of the appropriate levels must be injected during analysis of samples to monitor consistency of detector response. One standard analysis following three sample analyses is recommended once the standard curve has been established.

10.4.5
Chromatographic Conditions.

Initial Temperature	- 50 degrees C
Initial Hold Time	- 8 min
Program Rate	- 10 degrees C/min
Final Temperature	- 260 degrees C
Final Hold Time	- 7 min
Total Run Time	- 36 min
Injection Port Temperature	- 240 degrees C
FID Temperature	- 320 degrees C
Column Flow Rate	- 0.5 mL/min
Split Ratio	- 12:1
Injection Size	- 1 microliter

Column - 60 m X 0.25 mm ID
DB-5

10.5 Special Comments

10.5.1
Due to the nature of the samples, wash vials are a necessary part of the method. Do not overlook their use as autosampler corrosion and needle blockage will result.

11.0 CALCULATIONS

The previously noted Monsanto Data System uses the following formulae for data analysis:

$$\text{ACF= Area Correction Factor} = \frac{(\text{Amt C})}{(\text{Amt I})} \times \frac{(\text{Area I})}{(\text{Area C})}$$

Where Amt C = amount of component in a calibration sample, mg/L
Amt I = amount of internal standard in a calibration sample, mg/L
Area I = area of the internal standard chromatographic peak
Area C = area of the component chromatographic peak

Percent weight component =

$$(\text{ACF}) \times (100) \times \frac{(\text{Amt II})}{(\text{Amt CC})} \times \frac{(\text{Area CC})}{(\text{Area II})}$$

Where ACF = area correction factor
Amt II = amount of internal standard in a test sample, mg/L
Amt CC = amount of sample in a test sample, mg/L
Area CC = area of the component chromatographic peak
Area II = area of the internal standard chromatographic peak

12.0 DISCUSSION

12.1

Testing of this method was performed on twelve representative samples collected in 1983 and 1984. Consistent and reproducible results were observed in each sample for DCE.

12.2

It may be possible to extend this method to higher concentrations in CAC waste. This has been found to be unnecessary however. The determined concentration of DCE in the samples assayed by this method are significantly below the upper limit of validation in every case.

It may also be possible to extend this method to lower concentrations in CAC waste. The determined concentration of DCE in the samples assayed by this method are significantly higher than the lower limit of validation except for one outlier.

12.3

Toluene was chosen as the internal standard for this method because it is; 1) readily available, 2) highly pure (>99%), 3) available in large quantities, and 4) relatively inexpensive. It also has a desirable chromatographic characteristics, it elutes as a sharp peak in the center of the chromatogram in a region free of interferences.

13.0 REFERENCES

13.1

[a] J. W. Worley and J. C. Gee, "VALMET - A Useful Program for the Validation of Analytical Methods," unpublished.

[b] J. A. Morrell, "VALMET - A Computerized Program for the Analysis of Analytical Method Validation Data," MSL-2933; April 1983.

APPENDIX A

'or copies of the attachments listed here, contact the authors.

.. Raw Data Used in Valmet Analyses.

!. Example Chromatogram - Standard Injection

}. Sample Chromatogram - CAC Waste Sample
with Internal Standard Added.

ATTACHMENT 1

Raw Data Used in Valmet Analysis

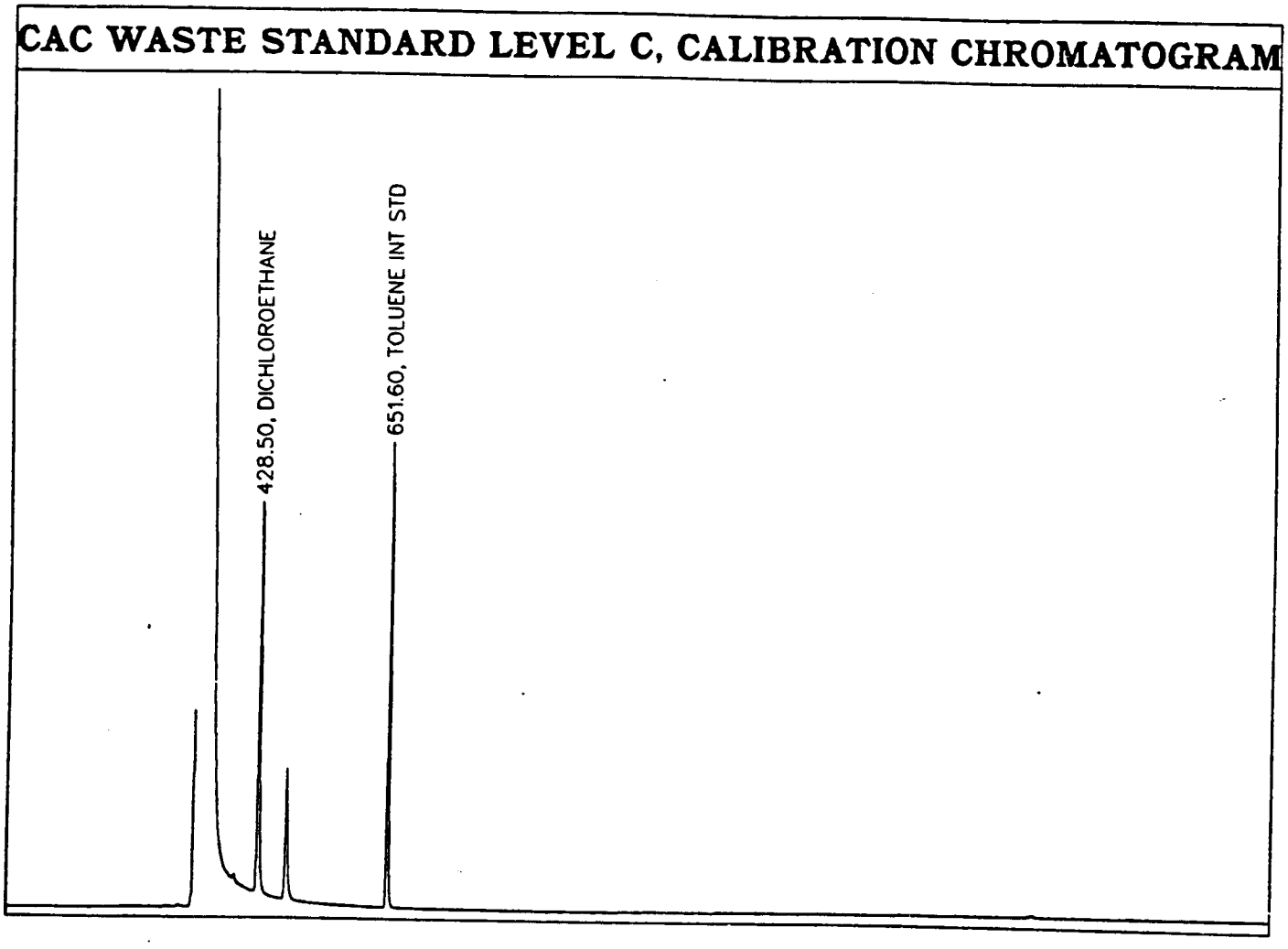
Component 1: 1,2-Dichloroethane
Number of levels: 4

	Level(1) = 1.150E-01	Number of Measurements = 8
1	9.914E-02	
2	9.998E-02	
3	1.007E-01	
4	1.037E-01	
5	1.044E-01	
6	9.473E-02	
7	9.918E-02	
8	9.709E-02	
	Level(2) = 6.776E-01	Number of Measurements = 8
1	6.663E-01	
2	6.661E-01	
3	6.655E-01	
4	6.759E-01	
5	6.651E-01	
6	6.676E-01	
7	6.601E-01	
8	6.664E-01	
	Level(3) = 2.275E+00	Number of Measurements = 8
1	2.266E+00	
2	2.258E+00	
3	2.245E+00	
4	2.260E+00	
5	2.260E+00	
6	2.259E+00	
7	2.251E+00	
8	2.235E+00	
	Level(4) = 7.171E+00	Number of Measurements = 8
1	7.155E+00	
2	7.113E+00	
3	7.077E+00	
4	7.097E+00	
5	7.080E+00	
6	7.112E+00	
7	7.124E+00	
8	7.132E+00	

Levels listed are in percent by weight units.

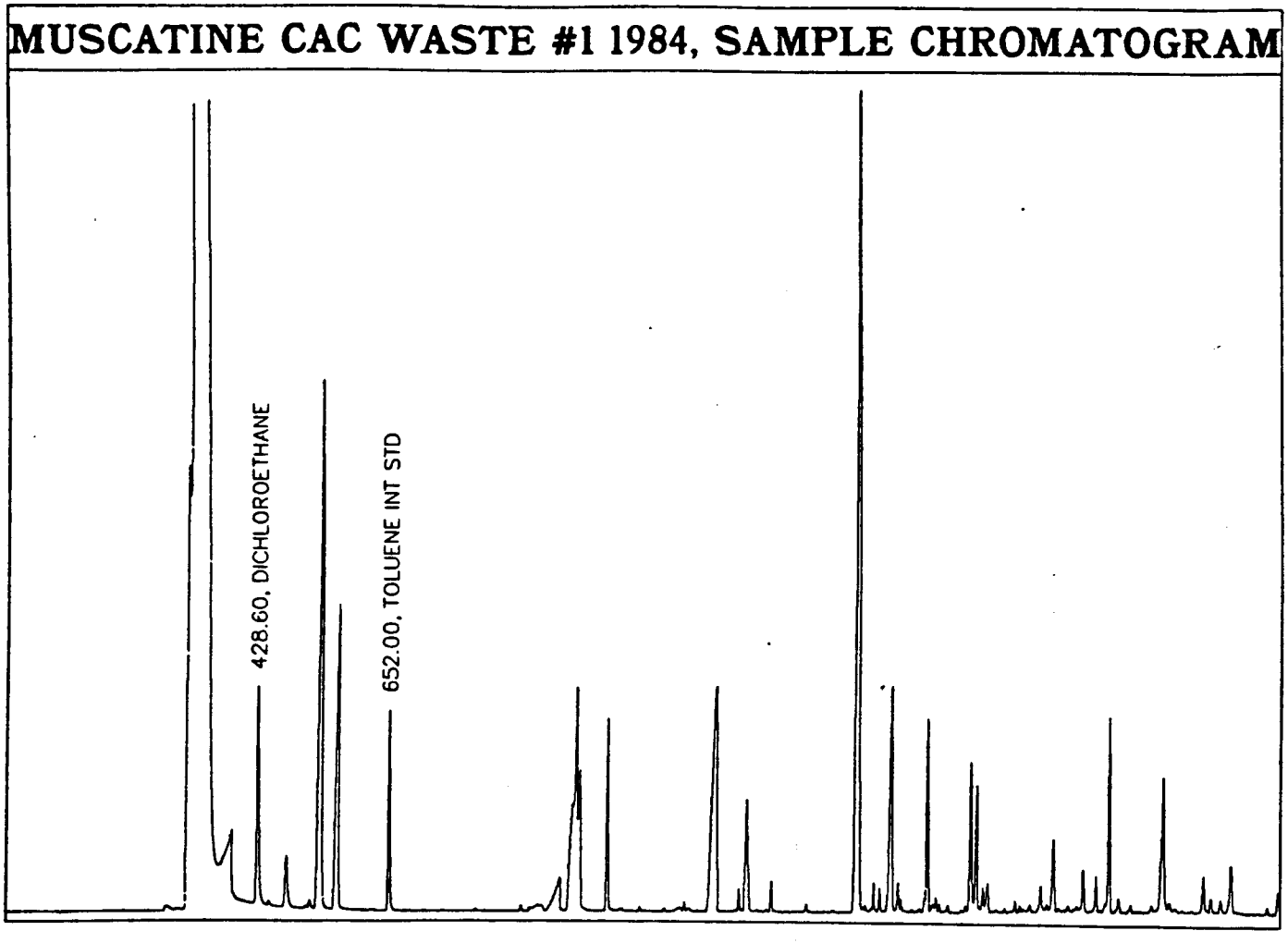
ATTACHMENT 2

Example Chromatogram - Standard Injection



ATTACHMENT 3

Sample Chromatogram - CAC Waste Sample
with Internal Standard Added



RESIDUE, FILTERABLE

Method 160.1 (Gravimetric, Dried at 180°C)

STORET NO. 70300

1. Scope and Application
   - 1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
   - 1.2 The practical range of the determination is 10 mg/1 to 20,000 mg/1.
2. Summary of Method
   - 2.1 A well-mixed sample is filtered through a standard glass fiber filter. The filtrate is evaporated and dried to constant weight at 180°C.
   - 2.2 If Residue, Non-Filterable is being determined, the filtrate from that method may be used for Residue, Filterable.
3. Definitions
   - 3.1 Filterable residue is defined as those solids capable of passing through a glass fiber filter and dried to constant weight at 180°C.
4. Sample Handling and Preservation
   - 4.1 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4°C, to minimize microbiological decomposition of solids, is recommended.
5. Interferences
   - 5.1 Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.
   - 5.2 Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180°C to insure that all the bicarbonate is converted to carbonate.
   - 5.3 Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200 mg.
6. Apparatus
   - 6.1 Glass fiber filter discs, 4.7 cm or 2.1 cm, without organic binder, Reeve Angel type 934-AH, Gelman type A/E, or equivalent.
   - 6.2 Filter holder, membrane filter funnel or Gooch crucible adapter.
   - 6.3 Suction flask, 500 ml.
   - 6.4 Gooch crucibles, 25 ml (if 2.1 cm filter is used).
   - 6.5 Evaporating dishes, porcelain, 100 ml volume. (Vycor or platinum dishes may be substituted).
   - 6.6 Steam bath.
   - 6.7 Drying oven, 180°C ±2°C.
   - 6.8 Desiccator.

Approved for NPDES
Issued 1971

6.9 Analytical balance, capable of weighing to 0.1 mg.

7. Procedure

7.1 Preparation of glass fiber filter disc: Place the disc on the membrane filter apparatus or insert into bottom of a suitable Gooch crucible. While vacuum is applied, wash the disc with three successive 20 ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Discard washings.

7.2 Preparation of evaporating dishes: If Volatile Residue is also to be measured heat the clean dish to 550 ±50°C for one hour in a muffle furnace. If only Filterable Residue is to be measured heat the clean dish to 180 ±2°C for one hour. Cool in desiccator and store until needed. Weigh immediately before use.

7.3 Assemble the filtering apparatus and begin suction. Shake the sample vigorously and rapidly transfer 100 ml to the funnel by means of a 100 ml graduated cylinder. If total filterable residue is low, a larger volume may be filtered.

7.4 Filter the sample through the glass fiber filter, rinse with three 10 ml portions of distilled water and continue to apply vacuum for about 3 minutes after filtration is complete to remove as much water as possible.

7.5 Transfer 100 ml (or a larger volume) of the filtrate to a weighed evaporating dish and evaporate to dryness on a steam bath.

7.6 Dry the evaporated sample for at least one hour at 180 ±2°C. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained or until weight loss is less than 0.5 mg.

8. Calculation

8.1 Calculate filterable residue as follows:

$$\text{Filterable residue, mg/l} = \frac{(A - B) \times 1{,}000}{C}$$

where:

A = weight of dried residue + dish in mg
B = weight of dish in mg
C = volume of sample used in ml

9. Precision and Accuracy

9.1 Precision and accuracy are not available at this time.

Bibliography

1. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 92, Method 208B, (1975).

RESIDUE, NON-FILTERABLE

Method 160.2 (Gravimetric, Dried at 103–105°C)

STORET NO. 00530

1. Scope and Application
   - 1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
   - 1.2 The practical range of the determination is 4 mg/1 to 20,000 mg/1.
2. Summary of Method
   - 2.1 A well-mixed sample is filtered through a glass fiber filter, and the residue retained on the filter is dried to constant weight at 103–105°C.
   - 2.2 The filtrate from this method may be used for Residue, Filterable.
3. Definitions
   - 3.1 Residue, non-filterable, is defined as those solids which are retained by a glass fiber filter and dried to constant weight at 103–105°C.
4. Sample Handling and Preservation
   - 4.1 Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result.
   - 4.2 Preservation of the sample is not practical; analysis should begin as soon as possible. Refrigeration or icing to 4°C, to minimize microbiological decomposition of solids, is recommended.
5. Interferences
   - 5.1 Filtration apparatus, filter material, pre-washing, post-washing, and drying temperature are specified because these variables have been shown to affect the results.
   - 5.2 Samples high in Filterable Residue (dissolved solids), such as saline waters, brines and some wastes, may be subject to a positive interference. Care must be taken in selecting the filtering apparatus so that washing of the filter and any dissolved solids in the filter (7.5) minimizes this potential interference.
6. Apparatus
   - 6.1 Glass fiber filter discs, without organic binder, such as Millipore AP-40, Reeves Angel 934-AH, Gelman type A/E, or equivalent.
     NOTE: Because of the physical nature of glass fiber filters, the absolute pore size cannot be controlled or measured. Terms such as "pore size", collection efficiencies and effective retention are used to define this property in glass fiber filters. Values for these parameters vary for the filters listed above.
   - 6.2 Filter support: filtering apparatus with reservoir and a coarse (40–60 microns) fritted disc as a filter support.

Approved for NPDES
Issued 1971

NOTE: Many funnel designs are available in glass or porcelain. Some of the most common are Hirsch or Buchner funnels, membrane filter holders and Gooch crucibles. All are available with coarse fritted disc.

6.3 Suction flask.

6.4 Drying oven, 103–105°C.

6.5 Desiccator.

6.6 Analytical balance, capable of weighing to 0.1 mg.

7. Procedure

7.1 Preparation of glass fiber filter disc: Place the glass fiber filter on the membrane filter apparatus or insert into bottom of a suitable Gooch crucible with wrinkled surface up. While vacuum is applied, wash the disc with three successive 20 ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Remove filter from membrane filter apparatus or both crucible and filter if Gooch crucible is used, and dry in an oven at 103–105°C for one hour. Remove to desiccator and store until needed. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg). Weigh immediately before use. After weighing, handle the filter or crucible/filter with forceps or tongs only.

7.2 Selection of Sample Volume

For a 4.7 cm diameter filter, filter 100 ml of sample. If weight of captured residue is less than 1.0 mg, the sample volume must be increased to provide at least 1.0 mg of residue. If other filter diameters are used, start with a sample volume equal to 7 ml/cm^2 of filter area and collect at least a weight of residue proportional to the 1.0 mg stated above.

NOTE: If during filtration of this initial volume the filtration rate drops rapidly, or if filtration time exceeds 5 to 10 minutes, the following scheme is recommended: Use an unweighed glass fiber filter of choice affixed in the filter assembly. Add a known volume of sample to the filter funnel and record the time elapsed after selected volumes have passed through the filter. Twenty-five ml increments for timing are suggested. Continue to record the time and volume increments until fitration rate drops rapidly. Add additional sample if the filter funnel volume is inadequate to reach a reduced rate. Plot the observed time versus volume filtered. Select the proper filtration volume as that just short of the time a significant change in filtration rate occurred.

7.3 Assemble the filtering apparatus and begin suction. Wet the filter with a small volume of distilled water to seat it against the fritted support.

7.4 Shake the sample vigorously and quantitatively transfer the predetermined sample volume selected in 7.2 to the filter using a graduated cylinder. Remove all traces of water by continuing to apply vacuum after sample has passed through.

7.5 With suction on, wash the graduated cylinder, filter, non-filterable residue and filter funnel wall with three portions of distilled water allowing complete drainage between washing. Remove all traces of water by continuing to apply vacuum after water has passed through.

NOTE: Total volume of wash water used should equal approximately 2 ml per cm^2. For a 4.7 cm filter the total volume is 30 ml.

7.6 Carefully remove the filter from the filter support. Alternatively, remove crucible and filter from crucible adapter. Dry at least one hour at 103–105°C. Cool in a desiccator and weigh. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg).

8. Calculations

8.1 Calculate non-filterable residue as follows:

$$\text{Non-filterable residue, mg/l} = \frac{(A - B) \times 1{,}000}{C}$$

where:

A = weight of filter (or filter and crucible) + residue in mg
B = weight of filter (or filter and crucible) in mg
C = ml of sample filtered

9. Precision and Accuracy

9.1 Precision data are not available at this time.

9.2 Accuracy data on actual samples cannot be obtained.

Bibliography

1. NCASI Technical Bulletin No. 291, March 1977. National Council of the Paper Industry for Air and Stream Improvement, Inc., 260 Madison Ave., NY.

United States
Environmental Protection
Agency

Environmental Monitoring and
Support Laboratory
Cincinnati OH 45268

Research and Development

EPA-600/4-84-017 Mar. 1984

EPA

Test Method
The Determination of Inorganic Anions in Water by Ion Chromatography — Method 300.0

James W. O'Dell, John D. Pfaff, Morris E. Gales, and Gerald D. McKee

1. Scope and Application

1.1 This method covers the determination of the following inorganic anions.

Analyte	Storet No. Total	Dissolved
Chloride	00940	—
Fluoride	00951	00950
Nitrate-N	00620	—
Nitrite-N	00615	—
Ortho-Phosphate-P	—	00671
Sulfate	00945	—

1.2 This is an ion chromatographic (IC) method applicable to the determination of the anions listed above in drinking water, surface water, and mixed domestic and industrial wastewater.

1.3 The Method Detection Limit (MDL, defined in Section 13) for the above analytes is listed in Table 1. The MDL for a specific matrix may differ from those listed, depending upon the nature of the sample.

1.4 This method is restricted to use by or under the supervision of analysts experienced in the use of ion chromatography and in the intrepretation of the resulting ion chromatogram. Each analyst must demonstrate the ability to generate acceptable results with this method, using the procedure described in Section 10.2.

1.5 When this method is used to analyze unfamiliar samples for any of the above anions, anion identification should be supported by the addition of spike solutions covering the anions of interest. The spike procedure is described in Section 11.6.

2. Summary of Method

2.1 A small volume of sample, typically 2 to 3 mL, is introduced into an ion chromatograph. The anions of interest are separated and measured, using a system comprised of a guard column, separator column, suppressor column, and conductivity detector.

3. Definitions

3.1 Stock standard solution — a concentrated solution containing a certified standard that is a method analyte. Stock standard solutions are used to prepare secondary standard solutions.

3.2 Calibration standards — a solution of analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare aqueous calibration solutions.

3.3 Quality control check sample — a solution containing known concentrations of analytes, prepared by a laboratory other than the laboratory performing the analysis. The analyzing laboratory uses this solution to demonstrate that it can

obtain acceptable identifications and measurements with a method.

3.4 Performance evaluation sample — a solution of method analytes distributed by the Quality Assurance Branch (QAB), Environmental Monitoring and Support Laboratory (EMSL-Cincinnati), USEPA, Cincinnati, Ohio, to multiple laboratories for analysis. A volume of the solution is added to a known volume of reagent water and analyzed with procedures used for samples. Results of analyses are used by the QAB to determine statistically the accuracy and precision that can be expected when a method is performed by a competent analyst. Analyte true values are unknown to the analyst.

3.5 Laboratory control standards — a solution of analytes prepared in the laboratory by adding appropriate volumes of the stock standard solutions to reagent water.

3.6 Laboratory duplicates — two aliquots of the same sample that are treated exactly the same throughout laboratory analytical procedures. Analyses of laboratory duplicates indicate precision associated with laboratory procedures but not the sample collection, preservation, or storage procedures.

3.7 Field duplicates — two samples taken at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.

4. Interferences

4.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or spiking can be used to solve most interference problems.

4.2 The water dip or negative peak that elutes near and can interfere with the fluoride peak can be eliminated by the addition of the equivalent of 1 mL of concentrated eluent (7.3 100X) to 100 mL of each standard and sample.

4.3 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

4.4 Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.

5. Safety

5.1 Normal, accepted laboratory safety practices should be followed during reagent preparation and instrument operation. No known carcinogenic materials are used in this method.

6. Apparatus and Materials

6.1 Balance — Analytical, capable of accurately weighing to the nearest 0.0001 g.

6.2 Ion chromatograph — Analytical system complete with ion chromatograph and all required accessories including syringes, analytical columns, compressed air, detector, and stripchart recorder. A data system is recommended for peak integration.

6.2.1 Anion guard column: 4 x 50 mm, Dionex P/N 030825, or equivalent.

6.2.2 Anion separator column: 4 x 250 mm, Dionex P/N 030827, or equivalent.

6.2.3 Anion suppressor column: fiber, Dionex P/N 35350, or equivalent.

6.2.4 Detector — Conductivity cell: approximately 6 μL volume, Dionex, or equivalent.

7. Reagents and Consumable Materials

7.1 Sample bottles: Glass or polyethylene of sufficient volume to allow replicate analyses of anions of interest.

7.2 Reagent water: Distilled or deionized water, free of the anions of interest. Water should contain particles no larger than 0.20 microns.

7.3 Eluent solution: Sodium bicarbonate (CAS RN 144-55-8) 0.003 M, sodium carbonate (CAS RN 497-19-8) 0.0024M. Dissolve 1.0081 g sodium bicarbonate ($NaHCO_3$) and 1.0176 g of sodium carbonate (Na_2CO_3) in reagent water and dilute to 4 liters.

7.4 Regeneration solution (fiber suppressor): Sulfuric acid (CAS RN 7664-93-9) 0.025N. Dilute 2.8 mL conc. sulfuric acid (H_2SO_4) to 4 liters with reagent water.

7.5 Stock standard solutions, 1000 mg/L (1 mg/mL): Stock standard solutions may be purchased as certified solutions or prepared from ACS reagent grade materials (dried at 105°C for 30 min.) as listed below.

7.5.1 Chloride (CL^-) 1000 mg/L: Dissolve 1.6485 g sodium chloride (NaCL, CAS RN 7647-14-5) in reagent water and dilute to 1 liter.

7.5.2 Fluoride (F^-) 1000 mg/L: Dissolve 2.2100 g sodium fluoride (NaF, CAS RN 7681-49-4) in reagent water and dilute to 1 liter.

7.5.3 Nitrate (NO_3^--N) 1000 mg/L: Dissolve 6.0679 g sodium nitrate ($NaNO_3$, CAS RN 7631-99-4) in reagent water and dilute to 1 liter.

7.5.4 Nitrite (NO_2^--N) 1000 mg/L: Dissolve 4.9257 g sodium nitrite ($NaNO_2$, CAS RN 7632-00-0) in reagent water and dilute to 1 liter.

7.5.5 Phosphate ($PO_4^{\equiv}$-P) 1000 mg/L: Dissolve 4.3937 g potassium phosphate (KH_2PO_4, CAS RN 7778-77-0) in reagent water and dilute to 1 liter.

7.5.6 Sulfate ($SO_4^{=}$) 1000 mg/L: Dissolve 1.8141 g potassium sulfate (K_2SO_4, CAS RN 7778-80-5) in reagent water and dilute to 1 liter.

7.5.7 Stability of standards: Stock standards (7.5) are stable for at least one month when stored at 4°C. Dilute working standards should be prepared weekly, except those that contain nitrite and phosphate should be prepared fresh daily.

8. Sample Collection, Preservation and Storage

8.1 Samples should be collected in scrupulously clean glass or polyethylene bottles.

8.2 Sample preservation and holding times for the anions that can be determined by this method are as follows:

Analyte	Preservation	Holding Time
Chloride	None required	28 days
Fluoride	None required	28 days
Nitrate-N	Cool to 4°C	48 hours
Nitrite-N	Cool to 4°C	48 hours
O-Phosphate-P	Filter and cool to 4°C	48 hours
Sulfate	Cool to 4°C	28 days

8.3 The method of preservation and the holding time for samples analyzed by this method are determined by the anions of interest. In a given sample, the anion that requires the most preservation treatment and the shortest holding time will determine the preservation treatment and holding time for the total sample.

9. Calibration and Standardization

9.1 Establish ion chromatographic operating parameters equivalent to those indicated in Table 1.

9.2 For each analyte of interest, prepare calibration standards at a minimum of three concentration levels and a blank by adding accurately measured volumes of one or more stock standards (7.5) to a volumetric flask and diluting to volume with reagent water. If the working range exceeds the linear range of the system, a sufficient number of standards must be analyzed to allow an accurate calibration curve to be established. One of the standards should be representative of a concentration near, but above, the method detection limit if the system is operated on an applicable attenuator range. The other standards should correspond to the range of concentrations expected in the sample or should define the working range of the detector. Unless the attenuator range settings are proven to be linear, each setting must be calibrated individually.

9.3 Using injections of 0.1 to 1.0 mL (determined by injection loop volume) of each calibration standard, tabulate peak height or area responses against the concentration. The results are used to prepare a calibration curve for each analyte. During this procedure, retention times must be recorded. The retention time is inversely proportional to the concentration.

9.4 The working calibration curve must be verified on each working day, or whenever the anion eluent is changed, and after every 20 samples. If the response or retention time for any analyte varies from the expected values by more than ± 10%, the test must be repeated, using fresh calibration standards. If the results are still more than ± 10%, an entire new calibration curve must be prepared for that analyte.

9.5 Nonlinear response can result when the separator column capacity is exceeded (overloading). Maximum column loading (all anions) should not exceed about 400 ppm.

10. Quality Control

10.1 Each laboratory using this method should have a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability (10.2) and the analysis of spiked samples as a continuing check on performance. The laboratory should maintain performance records to define the quality of data that are generated.

10.1.1 In recognition of the rapid advances occurring in chromatography, the analyst is permitted certain options to improve the separations or lower the cost of measurements. Each time such modifications to the method are made, the analyst is required to repeat the procedure in Section 10.2

10.1.2 The laboratory should spike and analyze a minimum of 10% of all samples to monitor continuing laboratory performance. Field and laboratory duplicates should also be analyzed.

10.2 Before performing any analyses, the analyst should demonstrate the ability to generate acceptable accuracy and precision with this method, using a laboratory control standard.

10.2.1 Select a representative spike concentration for each analyte to be measured. Using stock standards, prepare a quality control check sample concentrate in reagent water 100 times more concentrated than the selected concentrations.

10.2.2 Using a pipet, add 1.00 mL of the check sample concentrate (10.2.1) to each of a minimum of four 100-mL aliquots of reagent water. Analyze the aliquots according to the procedure in Section 11.

10.2.3 Calculate the average percent recovery (R), and the standard deviation(s) of the percent recovery, for the results.

10.2.4 Using the appropriate data from Table 2, determine the recovery and single operator precision expected for the method, and compare these results to the values calculated in Section 10.2.3. If the data are not comparable within control limits (10.3.1), review potential problem areas and repeat the test.

10.3 The analyst must calculate method performance criteria and define the performance of the laboratory for each spike concentration of analyte being measured.

10.3.1 Calculate upper and lower control limits for method performance as follows:

Upper Control Limit (UCL) = R + 3 s
Lower Control Limit (LCL) = R - 3 s

where R and s are calculated as in Section 10.2.3. The UCL and LCL can be used to construct control charts that are useful in observing trends in performance.

10.4 The laboratory should develop and maintain separate accuracy statements of laboratory performance for water and wastewater samples. An accuracy statement for the method is defined as R ± s. The accuracy statement should be developed by the analyses of four aliquots of water or wastewater, as described in Section 10.2.2, followed by the calculation of R and s.

10.5 Before processing any samples, the analyst must demonstrate through the analysis of an aliquot of reagent water that all glassware and reagent interferences are under control. Each time there is a change in reagents, a laboratory reagent blank must be processed as a safeguard against laboratory contamination.

10.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to monitor the precision of the sampling technique. When doubt exists over the identification of a peak in the chromatogram, confirmatory techniques such as sample dilution and spiking, must be used. Whenever possible, the laboratory should perform analysis of quality control check samples and participate in relevant performance evaluation sample studies.

11. Procedure

11.1 Table 1 summarizes the recommended operating conditions for the ion chromatograph. Included in this table are estimated retention times that can be achieved by this method. Other columns, chromatographic conditions, or

detectors may be used if the requirements of Section 10.2 are met.

11.2 Check system calibration daily and, if required, recalibrate as described in Section 9.

11.3 Load and inject a fixed amount of well mixed sample. Flush injection loop thoroughly, using each new sample. Use the same size loop for standards and samples. Record the resulting peak size in area or peak height units. An automated constant volume injection system may also be used.

11.4 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for a compound. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

11.5 If the response for the peak exceeds the working range of the system, dilute the sample with an appropriate amount of reagent water and reanalyze.

11.6 If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, spike the sample with an appropriate amount of standard and reanalyze.

Note: Retention time is inversely proportional to concentration. Nitrate and sulfate exhibit the greatest amount of change, although all anions are affected to some degree. In some cases, this peak migration can produce poor resolution or misidentification.

12. Calculation

12.1 Prepare separate calibration curves for each anion of interest by plotting peak size in area, or peak height units of standards against concentration values. Compute sample concentration by comparing sample peak response with the standard curve.

12.2 Report results in mg/L.

13. Precision and Accuracy — Method Detection Limit

13.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations listed in Table 1 were obtained using reagent water.

13.2 Single-operator accuracy and precision for reagent, drinking and surface water, and mixed domestic and industrial wastewater are listed in Table 2.

14. References

14.1 Annual Book of ASTM Standards, Part 31 Water, proposed test method for "Anions in Water by Ion Chromatography," p. 1485-1492 (1982).

14.2 Standard Methods for the Examination of Water and Wastewater, Method 400Z, "Anions by Ion Chromatography" proposed for the 16th Edition of Standard Methods.

14.3 Dionex, IC 16 operation and maintenance manual, PN 30579, Dionex Corp., Sunnyvale, California 94086.

14.4 Method detection limit (MDL) as described in "Trace Analyses for Wastewater," J. Glaser, D. Foerst, G. McKee, S. Quave, W. Budde, *Environmental Science and Technology,* Vol. 15, Number 12, p. 1426, December 1981.

Table 1. *Chromatographic Conditions and Method Detection Limits in Reagent Water*

Analyte	Retention[1] Time (Min)	Relative Retention Time	Method[2] Detection Limit mg/L
Fluoride	1.2	1.0	0.005
Chloride	3.4	2.8	0.015
Nitrite-N	4.5	3.8	0.004
O-Phosphate-P	9.0	7.5	0.061
Nitrate-N	11.3	9.4	0.013
Sulfate	21.4	17.8	0.206

Standard Conditions:

Columns — As specified in 6.2 *Sample Loop — 100 µL*
Detector — As specified in 6.2 *Pump Volume — 2.30 mL/Min*
Eluent — As specified in 7.3

[1] *Concentrations of mixed standard (mg/L)*

Fluoride 3.0 *O-Phosphate-P 9.0*
Chloride 4.0 *Nitrate-N 30.0*
Nitrite-N 10.0 *Sulfate 50.0*

[2] *MDL calculated from data obtained using an attenuator setting of 1 µMHO full scale. Other settings would produce an MDL proportional to their value.*

Table 2. *Single-Operator Accuracy and Precision*

Analyte	Sample Type	Spike (mg/L)	Number of Replicates	Mean Recovery %	Standard Deviation (mg/L)
Chloride	RW	0.050	7	97.7	0.0047
	DW	10.0	7	98.2	0.289
	SW	1.0	7	105.0	0.139
	WW	7.5	7	82.7	0.445
Fluoride	RW	0.24	7	103.1	0.0009
	DW	9.3	7	87.7	0.075
	SW	0.50	7	74.0	0.0038
	WW	1.0	7	92.0	0.011
Nitrate-N	RW	0.10	7	100.9	0.0041
	DW	31.0	7	100.7	0.356
	SW	0.50	7	100.0	0.0058
	WW	4.0	7	94.3	0.058
Nitrite-N	RW	0.10	7	97.7	0.0014
	DW	19.6	7	103.3	0.150
	SW	0.51	7	88.2	0.0053
	WW	0.52	7	100.0	0.018
O-Phosphate-P	RW	0.50	7	100.4	0.019
	DW	45.7	7	102.5	0.386
	SW	0.51	7	94.1	0.020
	WW	4.0	7	97.3	0.04
Sulfate	RW	1.02	7	102.1	0.066
	DW	98.5	7	104.3	1.475
	SW	10.0	7	111.6	0.709
	WW	12.5	7	134.9	0.466

RW = Reagent Water *SW = Surface Water*
DW = Drinking Water *WW = Wastewater*

*USGPO:1984-759-102-862

Appendix E

Field and Laboratory Data Sheets and Forms

TRAVERSE POINT LOCATION FOR CIRCULAR DUCTS

Plant _____

Date _____

Sampling location _____

Inside of far wall to outside of nipple _____

Inside of near wall to outside of nipple (nipple length) _____

Stack I.D. _____

Nearest upstream disturbance _____ dd

Nearest downstream disturbance _____ dd

Calculated by _____

SCHEMATIC OF SAMPLING LOCATION

TRAVERSE POINT NUMBER	FRACTION OF STACK I.D.	STACK I.D.	PRODUCT OF COLUMNS 2 AND 3 (TO NEAREST 1/8 INCH)	NIPPLE LENGTH	TRAVERSE POINT LOCATION FROM OUTSIDE OF NIPPLE (SUM OF COLUMNS 4 & 5)

CALCULATIONS FOR SELECTING A NOZZLE AND SETTING ISOKINETIC SAMPLING RATES

I. NOZZLE SELECTION

$$D_n = \left(\frac{T_s\ M_s}{\Delta P\ P_s}\right)^{0.25} \left(\frac{0.02678\ P_b}{(1-B_{wo})T_m C_p}\right)^{0.5}$$

T_s, °R	M_s	ΔP	P_s, in.Hg	P_b, in.Hg	B_{wo}→, decimal	$(1-B_{wo})$, decimal	T_m, °R	Cp	Dn, inches
					→				
					→				

Note: Approximate $\Delta H = \left(\frac{Dn\ selected}{Dn\ calculated}\right)^4 \times \Delta Ha$

II. K FACTOR FOR ISOKINETIC EQUATION

$$K = Dn^4\ Cp^2\ (1-B_{wo})^2\ (850.5\ \Delta Ha)\ \frac{M_d}{M_s}\ \frac{P_s}{P_m}$$

Dn, in.	Cp	B_{wo}→, decimal	$(1-B_{wo})$, decimal	ΔHa	M_d	M_s	P_s in.Hg	P_m in.Hg	K
		→							
		→							
		→							

III. ISOKINETIC ΔH CALCULATION

$$\Delta H = K\ \frac{\Delta P\ T_m}{T_s} \quad \text{for } T = °R$$

If desired, a calculator can be programmed to calculate ΔH during a test by storing the K value calculated above.

1. Program: GTO 000, LRN, 2nd Lbl, A, CLR, RCL, 1, x, (, R/S, +, 4, 6, 0,), ÷, (, R/S, +, 4, 6, 0,), x, R/S, =, R/S, LRN.
2. Store K in Register 01.
3. Press A, enter t_m, °F, press R/S, enter t_s, °F, press R/S, enter ΔP, press R/S, ΔH is displayed.
4. For next point, repeat step 3.

Note: To verify program let K = 10, t_m = 70°F, t_s = 250°F, ΔP = 1.0, →ΔH = 7.46

GAS VELOCITY AND VOLUMETRIC FLOW RATE

Plant and City ______________________ Date ____________

Sampling Location ______________________ Clock Time ____________

Run No. ______________________ Operator ____________

Barometric Pressure, in.Hg ____________ Static Pressure, in.H_2O ____________

Moisture, % ____________ Molecular wt., Dry ____________ Pitot Tube, Cp ____________

Stack Dimension, in. Diameter or Side 1 ____________ Side 2 ____________

FIELD DATA

TRAVERSE POINT NUMBER	VELOCITY HEAD (ΔP_s), in.H_2O	STACK TEMP., °F

CALCULATIONS

$M_s = Md \times (1 - \frac{H_2O}{100}) + 18 (\frac{H_2O\ \%}{100})$

$M_s = (\qquad) \times (1 - \frac{\qquad}{100}) + 18 (\frac{\qquad}{100})$

$M_s =$

$T_s = \qquad °F = \qquad °R = (°F + 460)$

$P_s = P_b + \frac{S.P.}{13.6} = (\qquad) + \frac{\qquad}{13.6}$

$P_s = \qquad$ in.Hg

$\overline{\sqrt{\Delta P}} =$

$V_s = 85.49 \times Cp \times \sqrt{\Delta P} \times \sqrt{\frac{T_s, °F}{P_s \times M_s}}$

$V_s = 85.49 \times (\qquad) \times (\qquad) \times \sqrt{\frac{\qquad}{\qquad \times \qquad}}$

$V_s = \qquad$ ft/s

$A_s = \qquad ft^2$

$Q_s = V_s \times A_s \times \frac{60\ s}{m}$

$Q_s = \qquad \times \qquad \times 60$

$Q_s = \qquad$ acfm

$Q_{s_{std}} = Q_s \times 17.647 \times \frac{P_s}{T_s} \times (1 - \frac{H_2O\ \%}{100})$

$Q_{s_{std}} = \qquad \times 17.647 \times \frac{\qquad}{\qquad} \times (1 - \frac{\qquad}{100})$

$Q_{s_{std}} = \qquad$ dscfm

DRY MOLECULAR WEIGHT DETERMINATION

PLANT _____

DATE _____ TEST NO _____

SAMPLING TIME (24-hr CLOCK) _____

SAMPLING LOCATION _____

SAMPLE TYPE (BAG, INTEGRATED, CONTINUOUS) _____

ANALYTICAL METHOD _____

AMBIENT TEMPERATURE _____

OPERATOR _____

ORSAT LEAK CHECKED _____

COMMENTS:

RUN / GAS	1		2		3		AVERAGE NET VOLUME	MULTIPLIER	MOLECULAR WEIGHT OF STACK GAS (DRY BASIS) M_d, lb/lb-mole
	ACTUAL READING	NET	ACTUAL READING	NET	ACTUAL READING	NET			
CO_2								44/100	
O_2 (NET IS ACTUAL O_2 READING MINUS ACTUAL CO_2 READING)								32/100	
CO (NET IS ACTUAL CO READING MINUS ACTUAL O_2 READING)								28/100	
N_2 (NET IS 100 MINUS ACTUAL CO READING)								28/100	
								TOTAL	

EMISSION TEST FIELD DATA

PLANT & CITY		SAMPLING LOCATION	SAMPLE T
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33	34 35 36 37 38 39 40 41	42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60 61 62 63 64 65	66 67 68 69 70 71 72 73 74 75 76 77 78 79 80
	/ /		

RUN NO.	OPERATOR	AMB. TEMP (°F)	BAR. PRESS. (IN. Hg)	STATIC PRESS (IN. H_2O)	FILTER NUMBER(S)	STACK INSIDE DIMEN. (INCHES)	PITOT TUBE Cp	THERM NO.	PAGE
1–6	7–27	28–30	31–34	35–39	40–57	58–69	70–73	74–76	77 78 79 80
			.	.		. .	.		0 F

PROBE LENGTH AND TYPE	NOZZLE I.D.	NOZZLE NO.	H_2O, (%)	SAMPLE BOX NO.	METER BOX NO.	METER Δ H @	METER CAL. FACTOR Y	LEAK CHECK IN. Hg	LEAK CHECK CFM	K FACTOR	C FACTOR	PROBE HEAT SET	BOX HEAT SET	REF. ΔP	RECORD DATA MINS
1–19	20–24	25–29	30–32	33–35	36–38	39–42	43–47	48–50	51–55	56–61	62–65	66–69	70–73	74–77	78–80
	.		.			.	.	.	.	.	.	.	.	.	.

TRAVERSE POINT NUMBER	SAMPLING TIME, min	CLOCK TIME (24 hr CLOCK)	GAS METER READING (V_m), ft^3	VELOCITY HEAD (Δp_s), in. H_2O	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O DESIRED	ORIFICE PRESSURE DIFFERENTIAL (ΔH), in. H_2O ACTUAL	STACK TEMPERATURE (T_s), °F	DRY GAS METER TEMPERATURE INLET ($T_{m_{in}}$), °F	DRY GAS METER TEMPERATURE OUTLET ($T_{m_{out}}$), °F	PUMP VACUUM, in. Hg	SAMPLE BOX TEMPERATURE °F	IMPINGER TEMPERATURE °F
7 8 9 10	11 12 13 . 14	15 16 17 18	19 20 21 . 22 23 24	25 26 27 28 29	30 31 . 32 33	34 35 . 36 37	38 39 40 41	42 43 44	45 46 47	48 49 50 . 51	52 53 54	55 56 57
	0		.									
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		
	.		.	.	.	.				.		

ISOKINETIC CALCULATION

SITE _____ TEST NO. _____

		RUN 1	RUN 2	RUN 3
1. Volume of dry gas sampled corrected to standard conditions. Note: V_m must be corrected for leakage if any leakage rates exceed L_a). $V_{m_{std}} = 17.65 \times V_m \times Y \left[\frac{P_{bar} + \frac{\Delta H}{13.6}}{T_m}\right] =$	V_m, ft^3			
	Y			
	P_{bar}, in.Hg			
	$\overline{\Delta H}$, $in.H_2O$			
	$\overline{T_m}$, °R			
	$V_{m_{std}}$, dscf			
2. Volume of water vapor at standard conditions, ft^3. $V_{w_{std}} = 0.04707 V_{lc} =$	V_{lc}, g			
	$V_{w_{std}}$, ft^3			
3. Moisture content in stack gas. $B_{ws} = \frac{V_{w_{std}}}{V_{m_{std}} + V_{w_{std}}} =$	B_{ws}			
	$1-B_{ws}$			
4. Dry molecular weight of stack gas, lb/lb-mole. $M_d = 0.440\ (\%\ CO_2) + 0.320\ (\%\ O_2) + 0.280\ (\%\ N_2 + \%\ CO) =$	% CO_2			
	% O_2			
	% N_2 + % CO			
	M_d, lb/lb-mole			
5. Molecular weight of stack gas. $M_s = M_d\ (1-B_{ws}) + 18\ B_{ws} =$	M_s, lb/lb-mole			
6. Stack velocity at stack conditions, fps. $V_s = 85.49\ Cp\ \left(avg.\ \sqrt{\Delta P}\right) \sqrt{\frac{T_s}{P_s M_s}} =$	P_{static}, $in.H_2O$			
	P_s, in.Hg			
	$\overline{T_s}$, °R			
	$\overline{\sqrt{\Delta P}}$			
	Cp			
	V_s, fps			
7. Isokinetic variation $\%\ I = \frac{V_{m_{std}} \times T_s \times 17.32}{V_s \times D_n^2 \times \theta \times P_s \times (1-B_{ws})}$	Dn, in.			
	θ, min.			
	% I			

VOST BLANK DATA

Corresponding Sample Runs _____

Sorbent Traps

1. Field Blanks	Pair No. 1	Pair No. 2	Pair No. 3
Date collected			
Time collected (24-h)			
Traps from Can No.			
Tenax Trap No.			
Tenax/charcoal Trap No.			
Connected to Sample Train No.			
Leak check, in.Hg/min at in.Hg vac.			
Culture tubes purged with N_2?			
Trap pair label ID			
Stored in Can No.			
Can Purged with N_2 and sealed?			
On Ice?			

2. Trip Blanks	Pair No. 1	Pair No. 2	Pair No. 3
Traps from Can No.			
Tenax/charcoal Trap No.			
Trap pair label ID			
Culture tubes remained closed?			
Stored in Can No.			
Can purged with N_2 and sealed?			
On Ice?			

3. Lab Blanks	Pair No. 1	Pair No. 2	Pair No. 3
Date traps removed from VOST prep room to sample storage			
Traps in Can No.			
Tenax Trap No.			
Tenax/charcoal Trap No.			
Trap pair label ID			
Culture tubes remained closed?			
Stored in Can No.			
Can purged with N_2 and sealed?			
On Ice?			

Water

Date collected _____ VOA bottle size _____ ml Label No. _____

Stored Can No. _____ Purged with N_2 and sealed? _____ On ice? _____

Water used for _____

_____

Remarks _____

_____

VOST SAMPLING DATA

Date _____ Location _____ Run No. _____ Operator _____

Test Condition _____ Sampling Train No. _____ Meter Console No. _____ Y-Factor _._ _ _

Trap Nos. _____ From Can No. _____ Pretest Leak Check _____ in.Hg/min at _____ in.Hg vacuum

Barometric Pressure (P_b) _ _._ _ in.Hg Ambient Temperature _____ °F Probe Purged ____ minutes at _____ liters/min

Sampling time, min.	Clock time (24-h)	Meter volume reading, liter	Rota-meter setting	Sampling train vacuum, in.Hg	Dry gas meter tempera-ture, °F	Primary condens-er exit temp., °F	Probe tempera-ture, °F		
0		_ _._ _ _							
_ _._									
	V_m	_ _._ _ _		Avg.	_ _ _._				

Post-Test Leak Check _____ in.Hg/min at _____ in.Hg Vacuum Trap Pair Label No. _____

Flow Direction Marked? _____ Condensate Recovered? _____ VOA Label Nos. _____

Total Volume of Condensate in Vials, _____ ml Remaining Condensate _____ mls Total Condensate Vol. _____ ml

Traps and Vial Stored in Can No. _____ on Ice? _____ Culture Tubes and Can Purged With N_2? _____

Comments:

Probe Leak Check Data:

Corresponding Blank Nos.

1) $V_{std} = V_m$, liters x Y x 17.647 x $\frac{P_b, \text{ in.Hg}}{T_m, {}^\circ R}$ = _ _._ _ _ liters

PARTICULATE SAMPLE RECOVERY AND INTEGRITY SHEET

Plant _____ Sample date _____

Sample location _____ Recovery date _____

Run number _____ Recovered by _____

Filter number(s) _____

MOISTURE

Impingers		Silica gel	
Final volume (wt)	_____ ml(g)	Final wt	_____ g
Initial volume (wt)	_____ ml(g)	Initial wt	_____ g
Net volume (wt)	_____ ml(g)	Net wt	_____ g
Description of impinger water	_____	_____	% spent

_____

Total moisture _____ g

RECOVERED SAMPLE

Filter container number(s) _____ Sealed _____

Description of particulate on filter _____

_____

Probe rinse container no.	_____	Liquid level marked	_____
_____ blank container no.	_____	Liquid level marked	_____
Impinger contents container no.	_____	Liquid level marked	_____
_____ blank container no.	_____	Liquid level marked	_____

Samples stored and locked _____

Remarks _____

_____

LABORATORY CUSTODY

Received by _____ Date _____

Remarks _____

_____

ROCESS SAMPLE AND INTEGRITY SHEET

Plant/City _____ Test/Run No. _____ Date _____

Sample		Container		Sample time (24-h)	Composite or grab	Preser-vation	Sampling person	Comments
Type/stream	Identification number	Type/size	Label No.					

Samples stored and locked _____

Date received by laboratory _____ Laboratory custodian _____

Remarks: _____

SAMPLING AND ANALYTICAL RECORD[a]

Traps used for:_____ Week of: _____

Trap pair Nos., T, TC	Date removed from lab	Date returned to lab	Use (sample or blank ID No.)	Current storage can No.	Can label No.	Date analyzed	Final status

Traps not used:

[a]Sampling team leader forwards sheet to sample custodian with copy to PC. Sample custodian gives sheet to analyst. Analyst forwards to PC each week. When all analyses are completed, original is returned to sample custodian, then PC.

NOZZLE CALIBRATION

Date_____ Calibrated by_____

Nozzle identification number	D_1, in.	D_2, in.	D_3, in.	ΔD, in.	D_{avg}

where:

$D_{1,2,3,}$ = nozzle diameter measured on a different diameter, in. Tolerance = measure within 0.001 in.

ΔD = maximum difference in any two measurements, in. Tolerance = 0.004 in.

D_{avg} = average of D_1, D_2, and D_3.

Figure E-1. Nozzle calibration data.

THERMOCOUPLE DIGITAL INDICATOR
AUDIT DATA SHEET

Date _____ Indicator No. _____ Operator _____

Test Point No.	Millivolt signal*	Equivalent temperature, °F*	Digital indicator temperature reading, °F	Difference, %
1				
2				
3				
4				

Percent difference must be less than or equal to 0.5%.

Percent difference:

$$\frac{(\text{Equivalent temperature } °R - \text{Digital indicator temperature reading } °R)(100\%)}{(\text{Equivalent temperature } °R)}$$

Where °R = °F + 460°F

* These values are to be obtained from the calibration data sheet for the calibration device.

ON-SITE AUDIT DATA SHEET

Audit Name: _____ Date: _____ Auditor: _____

Equipment	Reference	Reference Value	Value Determined	Deviation	Max. Allowable Deviation
Meter box inlet thermo.	ASTM-3F at ambient temp.				5°F
Meter box outlet thermo.	ASTM-3F at ambient temp.				5°F
Impinger thermometer	ASTM-3F at ambient temp.				2°F
Stack thermometer or Thermocouple	ASTM-3F at ambient temp.				7°F
	ASTM-3F at stack temp.				See table
Orsat analyzer	% O_2 in ambient air	20.8%			0.7%
Trip balance	IOLM std. weight				0.5 grams
Barometer	Corrected* NWS value				0.20 in. Hg

Reference temp. °F	32-140	141-273	274-406	407-540	541-673	674-760
Max. deviation °F	7	9	11	13	15	17

* Correction factor:

NWS value (in. Hg) - [Altitude (ft)/1000(ft/in. Hg)] + 0.74 in. Hg**

** 0.74 in. Hg is the nominal correction factor for the reference barometer against which the field barometer was calibrated.

If it is not feasible to perform the audit on any piece of equipment, record "N/A" in the space provided for the data.

FIELD AUDIT REPORT: DRY GAS METER BY CRITICAL ORIFICE

DATE: _____ CLIENT: _____

BAROMETRIC PRESSURE (P_{bar}): _____ in.Hg METER BOX NO. _____

ORIFICE NO. _____ PRETEST Y: _____ ΔH@ _____ in.H_2O

ORIFICE K FACTOR: _____ AUDITOR: _____

Orifice manometer reading ΔH, in.H_2O	Dry gas meter reading V_i/V_f, ft^3	Temperatures: Ambient T_{ai}/T_{af}, °F	Ambient Average T_a, °F	Dry gas meter Inlet T_{ii}/T_{if}, °F	Dry gas meter Outlet T_{oi}/T_{of}, °F	Dry gas meter Average T_m, °F	Duration of run Ø min.

Dry gas meter V_m, ft^3	$V_{m_{std}}$, ft^3	$V_{m_{act}}$, ft^3	Audit, Y	Y deviation, %	Audit ΔH@, in.H_2O	ΔH@ Deviation, in.H_2O

$$V_{m_{std}} = \frac{17.647(V_m)(P_{bar} + \Delta H/13.6)}{(T_m + 460)} = \quad ft^3$$

$$V_{m_{act}} = \frac{1203(\ \text{Ø}\ )(\ K\ )(P_{bar})}{(T_a + 460)^{1/2}} = \quad ft^3$$

$$\text{Audit Y} = \frac{V_{m_{act}}}{V_{m_{std}}} = \qquad \text{Y deviation} = \frac{\text{Audit Y - Pre-test Y}}{\text{Audit Y}} \times 100 =$$

$$\text{Audit } \Delta H@ = (0.0317)(\Delta H)(P_{bar})(T_m + 460)\left[\frac{\text{Ø}}{Y\,(V_m)(P_{bar} + \Delta H/13.6)}\right]^2 = \quad \text{in.}H_2O$$

Audit Y must be in the range, pre-test Y ±0.05 Y.
Audit ΔH@ must be in the range pre-test ΔH@ ±0.15 inches H_2O.

VOST CYLINDER GAS AUDIT DATA

Date _____ Location _____ Run No. _____ Operator _____

Test Condition _____ Sampling Train No. _____ Meter Console No. _____ Y-Factor _._ _ _

Audit Gas Cylinder No. _____ Cylinder Pressure, Initial _____ psig Final _____ psig

Outlet Pressure Setting _____ psig Manifold Purged _____ minutes at _____ liter/min Prior to Probe Purge

Trap Nos. _____ From Can No. _____ Pretest Leak Check _____ in.Hg/min at _____ in.Hg vacuum

Barometric Pressure (P_b) _ _._ _ in.Hg Ambient Temperature _____ °F Probe Purged ____ minutes at _____ liters/min

Sampling time, min.	Clock time (24-h)	Meter volume reading, liter	Rota-meter setting	Sampling train vacuum, in.Hg	Dry gas meter tempera-ture, °F	Primary condens-er exit temp., °F	Probe tempera-ture, °F	Audit gas rotameter setting	
0		_ _._ _ _							
_ _._									
V_m		_ _._ _ _		Avg.					

Post-Test Leak Check _____ in.Hg/min at _____ in.Hg Vacuum Trap Pair Label No. _____

Flow Direction Marked? _____ Culture Tubes and Can Purged With N_2? _____

Traps Stored in Can No. _____ Sealed? _____ On Ice? _____

1) $V_{std} = V_m$, liters x Y x 17.647 x $\frac{P_b, \text{ in.Hg}}{T_m, \text{ °R}}$ = _ _._ _ _ liters

INITIAL CEM CALIBRATION AND PERFORMANCE EVALUATION

Plant	_____	Pollutant	_____
Location	_____	Monitor	_____
Date	_____	Span	_____
Operator	_____	Chart scale	_____
PN	_____	Pbar, in.Hg	_____
		Tamb, deg. F	_____

Cal. gas conc., _____	Chart divisions: Direct injection to monitor	Chart divisions: Injection through system	Concentration predicted by equation*	Analyzer cal. error, % of span

COMMENTS: _____

_____

_____

* Perform linear regression of pretest chart divisions vs cal. gas concentrations to determine following equation:

Conc., _____ = chart divisions x (_____) + (_____)

Correlation coef. = _____

$$\text{Analyzer cal error} = \frac{[\text{Cal. gas conc.} - \text{conc. predicted}] \times 100}{\text{High cal. gas conc.}}$$

Minimum detectable limit = _____ or _____% of span

Response time to 95% of full scale _____s, _____s, _____s

Average response time _____s

Sampling system bias: Zero gas _____% of span

Mid gas (_____) _____% of span

$$\text{Sampling system bias} = \frac{[\text{Direct injection} - \text{pretest}]}{\text{Chart div. span}}$$

DAILY CEM CALIBRATION AND PERFORMANCE EVALUATION

Plant	_____	Pollutant	_____
Location	_____	Monitor	_____
Date	_____	Span	_____
Operator	_____	Chart scale	_____
PN	_____	Pbar, in.Hg	_____
Run No.	_____	Tamb, deg. F	_____

Cal. gas conc., _____	Chart divisions: Pretest	Chart divisions: Post-test	Concentration predicted by equation*	Analyzer cal. error, % of span	Drift, % of span

COMMENTS: _____

_____

_____

_____

_____

* Perform linear regression of pretest chart divisions vs cal. gas concentrations to determine following equation:

Conc., _____ = chart divisions x (_____) + (_____)

Correlation coef. = _____

$$\text{Analyzer cal error} = \frac{[\text{Cal. gas conc.} - \text{conc. predicted}] \times 100}{\text{High cal. gas conc.}}$$

$$\text{Drift} = \frac{[\text{Post-test} - \text{pretest}] \times 100}{\text{Chart div. span}}$$

Minimum detectable limit = _____ or _____% of span

Zero drift = _____% of span

Cal. drift = _____% of span

INITIAL CO CEM CALIBRATION AND PERFORMANCE EVALUATION

Plant	_____	Pollutant	Carbon monoxide
Location	_____	Monitor	_____
Date	_____	Span	_____
Operator	_____	Chart scale	_____
PN	_____	Pbar, in.Hg	_____
		Tamb, deg. F	_____

Cal. gas conc., _____	Chart divisions: Direct injection to monitor	Chart divisions: Injection through system	Concentration predicted by equation*	Linearity, % of span

COMMENTS: _____

_____

_____

* Perform linear regression of pretest chart divisions vs cal. gas concentrations to determine following equation:

Conc., _____ = chart divisions x (_____) + (_____)

Correlation coef. = _____

$$\text{Linearity} = \frac{[\text{Cal. gas conc.} - \text{conc predicted}] \times 100}{\text{High cal. gas conc.}}$$

Minimum detectable limit = _____ or _____% of span

Rise time to 90% of response by direct injection _____s, _____s, _____s

Average rise time _____s

Fall time to 90% of response _____s, _____s, _____s

Average fall time _____s

$$\text{Precision} = \frac{\text{Maximum difference} \times 100}{\text{Chart scale}}$$

Maximum diffe ence = individual - mean chart response for at least two repeated measurements of the same gas concentration

Precision = _____

DAILY CO CEM CALIBRATION AND PERFORMANCE EVALUATION

Plant _____ Pollutant _____

Location _____ Monitor _____

Date _____ Span _____

Operator _____ Chart scale _____

PN _____ Pbar, in.Hg _____

Run No. _____ Tamb, deg. F _____

Cal. gas conc., _____	Chart divisions Pretest	Chart divisions Post-test	Concentration predicted by equation*	Linearity, % of span	Drift, % of span

COMMENTS: _____

_____

_____

_____

* Perform linear regression of pretest chart divisions vs cal. gas concentrations to determine following equation:

Conc., _____ = chart divisions x (_____) + (_____)

Correlation coef. = _____

$$\text{Linearity} = \frac{[\text{Cal. gas conc.} - \text{conc. predicted}] \times 100}{\text{High cal. gas conc.}}$$

$$\text{Drift} = \frac{[\text{Post-test} - \text{pretest}] \times 100}{\text{Chart div. scale}}$$

Zero drift = _____% of span

Cal. drift = _____% of span

Minimum detectable limit = _____ or _____% of span

WASTE ANALYSIS PLAN

CAC THERMAL OXIDIZER INCINERATOR

MONSANTO CHEMICAL COMPANY - QUEENY PLANT

This Waste Analysis Plan describes the procedures that will be used to obtain chemical and physical data on the waste incinerated in the CAC Thermal Oxidizer to ensure proper treatment and maintain compliance with permit conditions.

The responsibility for implementation of the Waste Analysis Plan, as described herein, will lie with the Monsanto Chemical Company Queeny Plant Environmental Protection group.

I. FACILITY DESCRIPTION

The Monsanto Chemical Company Queeny Plant in St. Louis, Missouri is a manufacturer of a wide variety of specialty chemical products including industrial, agricultural and pharmaceutical chemicals, functional fluids, plasticizers and detergents. Alachlor, better known by its patent tradename Lasso$^{T.M.}$, and its major intermediate chloroacetyl chloride (CAC) are produced at the Queeny Plant.

The Alachlor process operates in response to seasonal market demands for the herbicide. It typically operates only in the first half of the year to produce enough product for seasonal farmer market demands, and then shuts down for the second half of the year. Accordingly, the CAC Incinerator which treats the wastes produced in the manufacture of Alachlor also operates only when the processes operate. Two waste streams are burned in the CAC Incinerator from the CAC Intermediate and Alachlor processes -- CAC Waste and Azomethine (Azo) Residue. No off-site or ex-process wastes are incinerated in the CAC incinerator.

CAC waste (D002, D003) results from impurities formed in the process chlorinator which are separated in the light ends column and solvent columns. Both separation columns are operated continuously with the waste stream fed continuously to the CAC waste feed tank (12,500 gallon capacity). The combined CAC waste streams are fed to the incinerator through a continuously circulating line.

The Azo Residue (nonhazardous) is a column bottoms waste stream from one of the three manufacturing steps in the Alachlor process. The Azo Residue is an extremely viscous material and, in order to facilitate pumping from the bottoms, No. 2 fuel oil is added to keep it in a fluid state. The waste is pumped to the Azo Residue tank (6,000 gallons capacity) which feeds the CAC incinerator.

II. OBJECTIVE AND APPROACH

The objective of this waste analysis plan is to outline the sampling and analysis program elements developed to confirm that the waste types we treat in the CAC incinerator meet the chemical and physical characteristics described in our RCRA Part B permit. Permit compliance leads to safe operation and, in turn, protection of human health and the environment.

The development of this waste analysis plan began with the defining the hazardous wastes to be treated in the incinerator, using the approach shown in Figure 1. Defining each waste stream involves identifying the waste characteristics that are necessary to treat the hazardous waste effectively and the treatability limitations of the CAC waste.

In order to ensure that the CAC waste remains within our permit conditions, periodic waste analyses (recharacterizations) will be performed. Our planning therefore included selection of the parameters that best characterize CAC waste and the frequency of periodic analyses.

The sampling and analysis methods for the waste parameters are specified in the plan, and quality assurance/quality control (QA/QC) procedures are established for these methods.

III. IDENTIFICATION OF WASTE TYPES

Table 1 lists the characteristics of the CAC waste treated in the incinerator. The data reflects analytical results from past and current samples of waste. The waste characterizations were performed by our on-site process QC laboratory, corporate headquarter research laboratory, and an offsite commercial laboratory. An ongoing program is underway to update and/or confirm waste characterization data for permit compliance. A collection of analytical results and reports from these laboratories will be attached in Appendix A. Quality assurance and quality control programs associated with the commercial laboratories are described in Section VI.C.

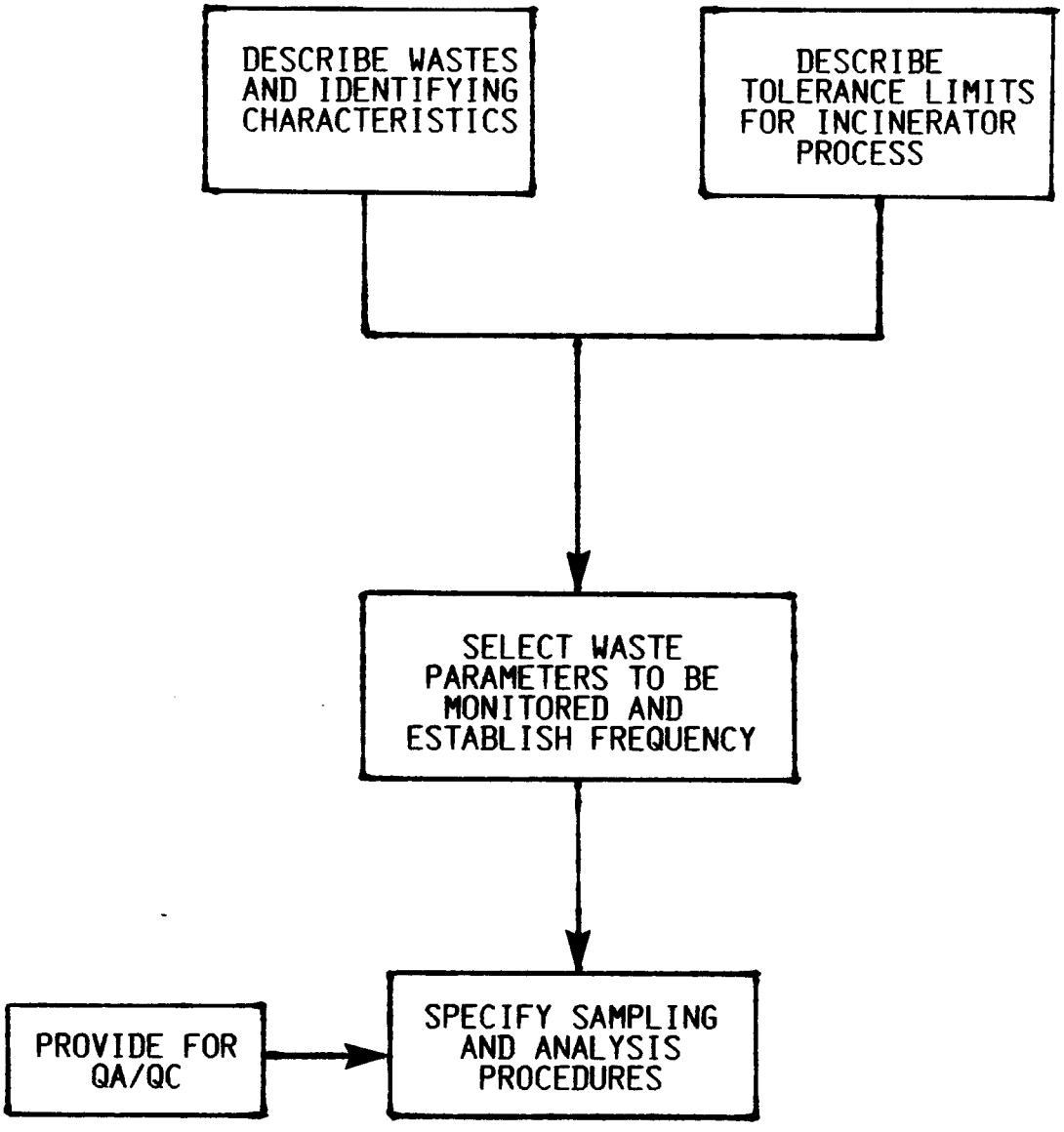


Figure 1. Approach to waste analysis plan development.

TABLE 1. CAC THERMAL OXIDIZER - WASTE CHARACTERISTICS

Waste Type	EPA Waste Number	Physical Properties	Chemical Composition
CAC Residue	D002, D003	sp.gr.= 1.44 ash = Btu = 8260/lb viscosity = 1.56 cS black liquid with strong odor	34.5% Acetyl chloride 18.8% Dichloroacetic anhydride 18.5% High boiling tars 11.1% Chloroacetyl chloride (CAC) 7.1% 2-Chloroethyl-4-chlorobutyrate 3.3% Acetic anhydride 2.4% Butyroacetone 2.0% Acetic acid 0.2% 1,2-Dichloroethane 0.5% Propargyl chloride

The following boundary conditions have been established for the CAC Residue (D002, D003) characteristics:

* +/- 50 percent of the viscosity listed in Table 1,

* Ash content above 0.3 %,

* Organically bound chloride content above 40 %.

Any waste feed that exceeds the boundary conditions will be handled according to the procedures described in Section V, "Parameters to be Monitored." Our experiences with this waste stream and the process that generates it has led us to establish these conditions, and we do not expect the waste stream to vary outside these boundaries.

IV. INCINERATOR TOLERANCE LIMITS

The waste feed to the CAC incinerator must be a pumpable liquid for efficient steam atomization and have a heating value that meets the temperature requirements of the CAC incinerator. The total feed rate to the incinerator must range from 9.1×10^6 Btu/hr to 14.1×10^6 Btu/hr, or as set in the final RCRA Part B permit conditions. The heating value of each organic constituent in the waste feed(s) must be greater than the heating value for the pure indicator POHC designated at the trial burn -- tetrachloroethylene (1.19 kcal/g or 2142 Btu/lb), which must attain at least a 99.99 destruction and removal efficiency during the trial burn. The chloride content of the waste feed must not exceed 40 percent. This limit leads to optimum scrubber removal of HCl emissions. This value is the maximum concentration for which compliance with CAC incinerator performance standards will be demonstrated during the trial burn. Ash content of the waste feed must be less than 0.3 percent in order to comply with particulate emissions standards.

Although the CAC waste is segregated by means of the storage tank and dedicated piping a review of waste compatibilities was undertaken using the procedures described in A Method for Determining the Compatibility of Hazardous Wastes, EPA-6--/2-80-076, April 1980. The method is useful in the management of hazardous wastes, in that it determines the types of wastes which should not be mixed (or stored in close proximity in the event of an accidental release) and predicts the adverse reaction consequences that can inflict damage to life, property and the environment. The method consists of a step-by-step compatibility analysis procedure and a compatibility chart. The chart is the key element in the use of the method. Wastes which could be mixed (in the event of an accident) are first subjected, through the compatibility analysis procedure, to identication and classification, and the chart is used to predict the

compatibility of the wastes upon the remote chance of mixing. Waste reactivity group classifications to be burned at the Thermal Oxidizer facility include:

1. Water reactive substances (acetyl chloride and chloroacetyl chloride)
2. Halogenated organics (acetyl chloride, ethylene dichloride, chlorobutyroacetones)
3. Combustible materials (No. 2 fuel oil).

A cross check of all possible combinations of the above groups in the chart (Figure 6 in EPA-600/2-80-076) shows that no incompatiblities result from mixture as long as the CAC Residue waste stream remains isolated until the flame zone. CAC waste and Azo Residue are burned at separate times in the incinerator.

V. PARAMETERS TO BE MONITORED

The parameters for which the CAC incinerator waste feed will be analyzed are identified in Table 2 along with the rationale for selecting each parameter.

Recharacterization of the CAC waste is scheduled for an annual frequency. The nature of the chemical process generating CAC waste is quite consistent because of the need for consistent product quality, the experience in operating the process for many years, and the seasonal "campaigns" of the process to meet market demands.

If recharacterization analyses ever indicate that a waste is beyond the parameter bounds established, we will conduct a reanalysis to resolve the rejection. We will also reanalyze a waste for the applicable parameter(s) should our process area personnel notify us that their process operation has changed and may affect the waste composition or physical properties. Upon confirming a parameter change beyond our permit conditions, the waste will remain stored in its tank until the proper permit modification (minor or major) has been obtained.

TABLE 2. WASTE ANALYSIS PARAMETERS

Waste Stream	Parameters	Rationale for Parameters
CAC	Heat Value	Required under 40 CFR 270.62 and to check for significant variations in composition
CAC	Viscosity	Required under 40 CFR 270.62 and to ensure adequate atomization
CAC	Volatile Organic Constituents	Identify any POHCs that are present to determine if Heat of Combustion values exceed tetrachloroethylene's
CAC	Organochlorine Content	To maintain compliance with HCl emissions standard required under 40 CFR 264.343(b)
CAC	Ash	To maintain compliance with particulate emissions standard required under 40 CFR 264.343(c)

VI. WASTE SAMPLING AND ANALYSIS

A. Sampling

The CAC Residue is sampled from its pipeline leading to the CAC incinerator. The CAC Residue feed is sampled just after the flow meter and control valve, enroute to the burner gun, as shown in Figure 2. The sampling location is controlled by a valve/spigot which points down.

The CAC waste feed line, when operating, is pumped in constant recycle to keep the feed mixed and as homogenous as possible. Therefore, composite sampling is not necessary to collect a "representative" sample. Furthermore, the personal protection precautions necessary to collect waste feed samples preclude the collection of multiple samples and extra handling to create composite samples. Table 3 presents the departmental hazardous chemical communication program information for the safe handling of the CAC waste feed sample (Note: CAC Residue is presented as chloroacetyl chloride since it contains both acetyl chloride and CAC).

The following procedure is to be used for collecting waste samples in storage containers for analysis:

1. Consider the properties and hazards (i.e., reactivity, composition, volatility, etc.) of the waste stream before sampling. Consult the departmental hazardous chemical information before attempting to collect a sample. Choose the sample container that is made of material which is nonreactive to the waste sample, of adequate wall thickness, and large enough to contain the optimum sample volume for the desired analysis (typically, this will be a 1 or 2 liter glass bottle or jar with teflon-lined lid).

2. Position the sample container under the line tap, and slowly open the sample line valve to allow the sample to flow into the container without splashing or spilling. Close the sample line valve just before enough sample is collected. Check to see that the sample line line valve is completely closed.

3. After sampling, cap the container.

4. Label each of the sample containers following SW-846 (3rd Edition) section 9.2.2.7 protocol. Record sampling data (e.g., time, location, date, volume of sample, method, observations, etc.) on the field data sheet or in a field logbook following the sample collection. Seal each container with stretch tape around the lid in such a way that it is necessary to break the seal in order to open the container.

5. Fill out a chain-of-custody record form following SW-846 section 9.2.2.7 protocol for each sample set destined to a particular laboratory for analysis.

Figure 2. Schematic diagram of CAC waste sampling location.

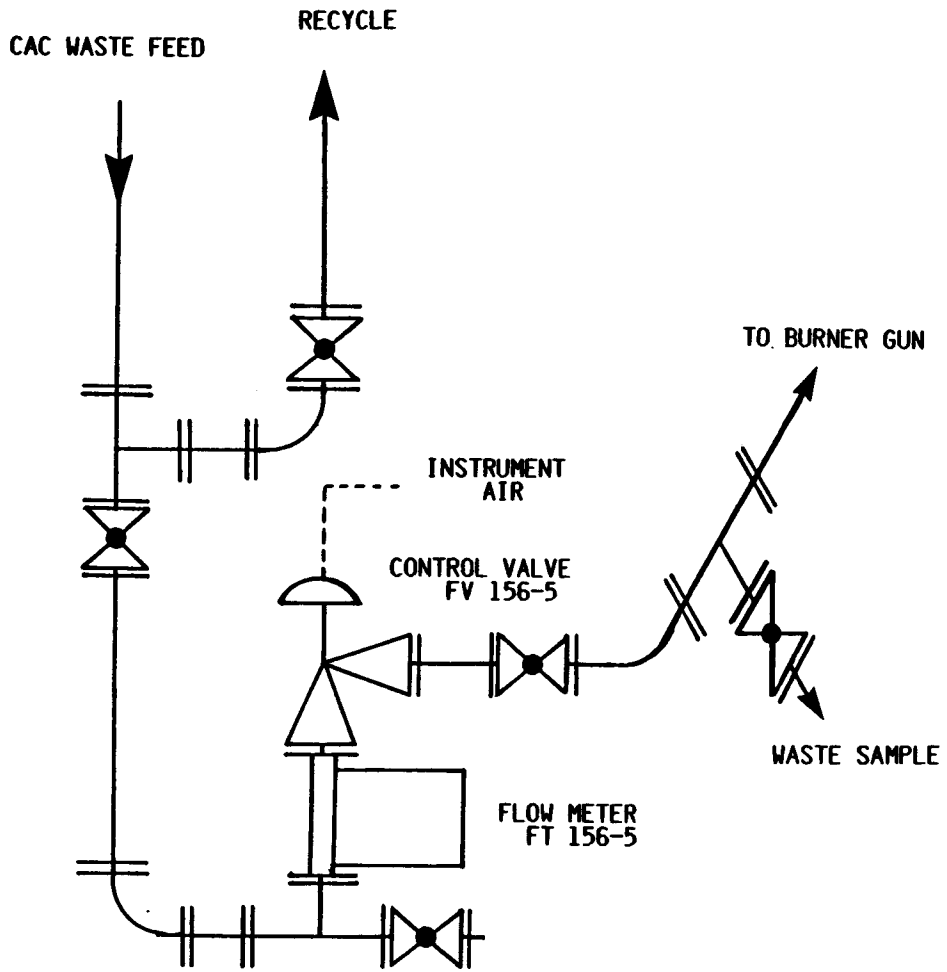


TABLE 3. HANDLING INSTRUCTIONS FOR CAC RESIDUE TO MINIMIZE PERSONAL EXPOSURES

Chemical Name	Hazards	First Aid	Handling Instructions
Chloroacetyl chloride CAS#000079049	Corrosive to skin and eyes. Very reactive with water.	Remove to fresh air. Wash skin and eyes with water for 15 minutes while removing contaminated clothing. Report immediately to the dispensary for further treatment.	All spills are to be washed with large amounts of water. Impervious(PVC) gloves and white disposable coveralls in conjunction with a full face respirator equipped with yellow organic vapor/acid gas cartridges must be worn when breaking into lines or handling in the open.

B. Analysis

Table 4 identifies the analytical methods to be employed to measure each waste parameter. The test methods were chosen from EPA-SW-846, Test Methods for Evaluating Solid Waste, Physical/ Chemical Methods (Third Edition), from the American Society for Testing and Materials (ASTM) compendium of test methods, and from EPA-600/8-84-002, Sampling and Analysis Methods for Hazardous Waste Combustion.

TABLE 4. SUMMARY OF WASTE ANALYSIS METHODS FOR RECHARACTERIZATION PARAMETERS

Parameter	Preparation Method	Analytical Method
Heat Value	N/A	ASTM D240 (Bomb Calorimeter)
Viscosity	N/A	ASTM D445 (Kinematic viscometer)
Ash	N/A	ASTM D482 (Gravimetric loss on ignition)
Organochlorine Content	EPA 3580 (Dilution)	EPA 9020 (Microcoulometry)
Organics	EPA 3580	EPA 8240 (GC/MS)
Ethylene dichloride		
Wide Scan - others		

C. Quality Assurance/Quality Control (QA/QC)

Monsanto's QA/QC program for the waste sampling and laboratory analysis serves to document the quality (i.e., accuracy and precision) of generated data; maintain the quality of data within predetermined tolerance limits for specific sampling and analytical procedures; and provide guidelines for corrective action if a procedure is not within control limits. This program will be implemented for subsequent waste sampling and analysis operations related to the CAC Thermal Oxidizer. The program's goal is to obtain accurate and precise waste characterization data so that we can assure that the wastes we incinerate possess the chemical/physical properties specified in our permit.

1. Responsibility - Responsibility for the management of the QA/QC program rests with the Environmental Protection group of the Monsanto Chemical Company's Queeny Plant.

2. QC Objectives - Laboratory objectives for QA/QC are summarized below:

   Accuracy and Precision - To achieve accuracy goals, external standards for organic constituents and choride analyses of reagent grade quality are used for calibration and spiking. Control limits for QC spike accuracy and duplicate precision have been established for EPA Method 8240 and for individual compounds as a function of spike concentration. These goals will be maintained for all applicable parameters.

   Completeness - Analytical results will be obtained for at least 95% of the collected samples.

   Representativeness - Selection of sampling sites, frequency, procedures, and sampling equipment/containers are directed at obtaining the most representative sample possible and are based on standardized practices.

3. Sampling and Analytical Procedures - Sections VI.A and VI.B of this Waste Analysis Plan detail the sampling and analytical procedures (by reference) and frequency of tests to be run for the incineration waste samples. These procedures are well documented and will be kept on file at the plant.

4. Sample Custody and Documentation - Chain-of-custody procedures will be the responsibility of the plant's Environmental Protection group, who will act as sample custodian at the plant. Signed custody records which document the sample path from plant to a particular laboratory will be maintained at the plant. Also

   maintained at the plant will be a log of all field

samples taken and the filing in Appendix A of this Waste Analysis Plan of all waste recharacterization data. The outside commercial laboratory, PEI Associates Inc., maintains a QA/QC program and CLP certification which includes procedures for numbering and documenting sample path through the lab, destiny of remaining sample after analysis, and forwarding test result documents to management.

5. Calibration Procedures and Frequency - the calibration procedures and frequency for the analytical instruments will be set hierachically according to SW-846 procedures, the laboratory's own QA/QC program, or manufacturer's specifications. Instrument calibration information is retained at each laboratory in logbooks using forms as described in SW-846 Section 1.5 quality control documentation.

6. Internal QC Checks - Blind duplicate samples submitted to the outside laboratory will serve as an internal QC check. Blank analytical samples are run as part of the SW-846 organic procedures.

7. Audits, Corrective Action, and QA Reports to Management - The outside laboratory participates in external performance audit programs which include the quarterly blind audit samples required for participation in the USEPA Contract Laboratory Program (CLP). Systems audits are conducted on a random basis by the lab's Quality Assurance Coordinator and are summarized in a written report available for review. Corrective action is initiated based on poor method recoveries or precision as outlined in SW-846, poor performance or systems audit results, data completeness below required limits, or from failure to adhere to the QA Plan or SOPs. All final analytical reports are reviewed for technical aspects and quality assessment according to written laboratory guidelines by several layers of management before final approval and issuance.

APPENDIX A - ANALYSIS RESULTS

FILES - WASTE # 001 CAC Cl. WASTE ;
" # 005 HERBICIDE WASTE

YC - DON LYNSKEY
GAYLE MALCOLMEY

Monsanto

FROM (NAME—LOCATION—PHONE) D. L. Kleyer - U3I - (4-8461)

DATE: August 20, 1984

SUBJECT: RCRA Incinerator Application

REFERENCE:

cc: H. C. Berk - U3I
J. R. Klieve - 1630
J. M. Warner - U3I
L. Weitzman - Acurex
J. W. Worley - O3C

TO: J. C. Fowler - 1630

The following information will be covered in this memo: Heating value of wastes, physical description, identification of waste and quantification of waste components for both chloroacetyl chloride and canning plant waste streams.

CAC WASTE

This waste is best described as a black colored liquid with a strong odor. The following components were identified and quantitated in samples of this waste taken during June and July of 1984. Other components can be best described as chlorinated esters having a molecular weight greater than 200 of a type similar to 2-chloroethyl-4-chlorobutyrate.

SAMPLE , level in wt. %

Component	1	2	3	4	5	ave.
Acetyl Chloride	*37.7	24.4	36.8	38.9	<1	27.6 (34.5 ± 6.8)
Propargyl Chloride	0.68	0.28	0.49	0.56	<0.1	0.40 (0.50 ± 0.17)
Butyrolactone	2.66	3.29	2.05	1.76	30.5	8.05 (2.4 ± 0.7)
2-Chloroethyl-4-chlorobutyrate	7.76	8.89	6.70	5.11	18.4	9.37 (7.1 ± 1.6)
Acetic Acid	3.06	1.17	2.33	1.47	<0.5	1.61 (2.0 ± 0.9)
1,2-Dichloroethane	*1.89	1.35	2.01	1.89	<0.5	1.43 (1.79 ± 0.3)
Acetic Anhydride	3.36	2.94	3.36	3.55	<0.5	2.64 (3.3 ± 0.3)
Chloroacetyl Chloride	12.0	11.8	9.3	11.4	1.38	9.18 (11.1 ± 1.2)
High Boiling Tars / ~~Closure~~ Dichloroacetic anhydrid	69.1	54.1	63.0	64.6	50.3	60.2 (18.5 ~~18.5~~ 18.8)
		level in ppm				
chloromethane	*68	127	83	72	<25	70 (88 ± 27)
dichloromethane	*30	57	25	28	<25	28 (35 ± 15)
chloroform	*100	156	102	148	137	129 (127 ± 30)

Carbon Tetrachloride	*47	60	112	48	<25	53	67±31
Tetrachloroethane	*32	39	45	31	<25	29	37±7
2,3-Dichloro-1-propene	*118	473	649	529	664	487	442±228
1,2,3Trichloropropene	*234	356	1029	379	431	486	500±359

* denotes an appendix VIII hazardous constituent

Elemental analysis was also performed. Results are listed below.

Sample	% C	% H	% Cl
1	33.14	3.71	40.85
2	34.99	3.92	37.65
3	34.94	3.96	37.57
4	34.95	3.98	37.52
5	38.08	4.19	32.30
average	35.22	3.95	37.18

34.5 ±0.9

3.9 ±0.1

38.4 ±1.6

Heat of combustion and viscosity were also determined for these samples.

Sample	Heat of Combustion	Viscosity (25% C)
1	8283 Btu/lb	1.530 centiStokes
2	8049	2.041
3	8308	1.332
4	8394	1.354
5	7290	7.310
average	8065	2.713

8260

1.56 ±0.3

PEI Analytical Services

CLIENT: Terran Corporation
P.O. Box 1410
Fairborn, Ohio 45324-6345

ATTN: Mr. Jim Peters

PROJECT: 4874
REQ: T8-04-122
RECEIVED: 4/19/88
FILE: 670

DATE REPORTED: 6/7/88

MATRIX: Wastes

Sample ID PEI No.	BLANK	AZO RESIDUE -01	CAC RESIDUE -02
Analyte			
1,2-Dichloroethane	ND(20 Ug/G)	ND(20 Ug/G)	2.39 Mg/G
Tetrachloroethene	ND(20 Ug/G)	ND(20 Ug/G)	0.31 Mg/G
Other compounds Tentatively Identified			
Dichloromethane		240 Ug/G	
Cycloalkane		420 Ug/G	
Acetic acid, methyl ester			67 Mg/G
Chloroacetic acid, methyl ester			4 Mg/g

PEI Associates, Inc.
11499 Chester Rd.
Cincinnati, Ohio 45246
513-782-4700

Approved By:

H. William Jess
Organic Laboratory
Supervisor

PEI Analytical Services

Analytical Notes
Terran Project
PN 4874 TB-04-122 Received 4/19/88

Initially, a 2ul aliquot of AZO residue was to be measured with a 10ul syringe and added to a 250ml vol. flask filled to the mark with organic free water. The AZO material proved to viscous to draw into a syringe.

An aliquot of the AZO material was then weighed into a 10ml vol. flask filled with water. The AZO did not react with the water, but floated on the surface. Analysis of aliquots of the water showed no detectable concentrations of the analytes of interest.

A 25ml vol flask was then filled to the mark with methanol, and 92.1 mg of AZO material was weighed into the flask. The concentration of this solution was then 3.684 mg of AZO material / 1ml methanol. Aliquots of this solution were added to 5ml of organic free water and analyzed according to USEPA method 8240. (Purge and Trap, Methanolic dilution). The samples were analyzed for all HSL volatiles. In addition, all other peaks were compared to the EPA/NIH library of mass spectra to attempt to identify any other compounds present.

Aliquots of the CAC residue were also added to water. Again, analysis of the aqueous phase showed no detectable levels of organic compounds of interest. A solution of the CAC was then made up in methanol (2.08 mg/ml) and aliquots of the methanolic solution were analyzed in the same manner as the AZO residue.

PEI Associates, Inc.

VOLATILE SURROGATE RECOVERY SUMMARY
GC/MS Laboratory

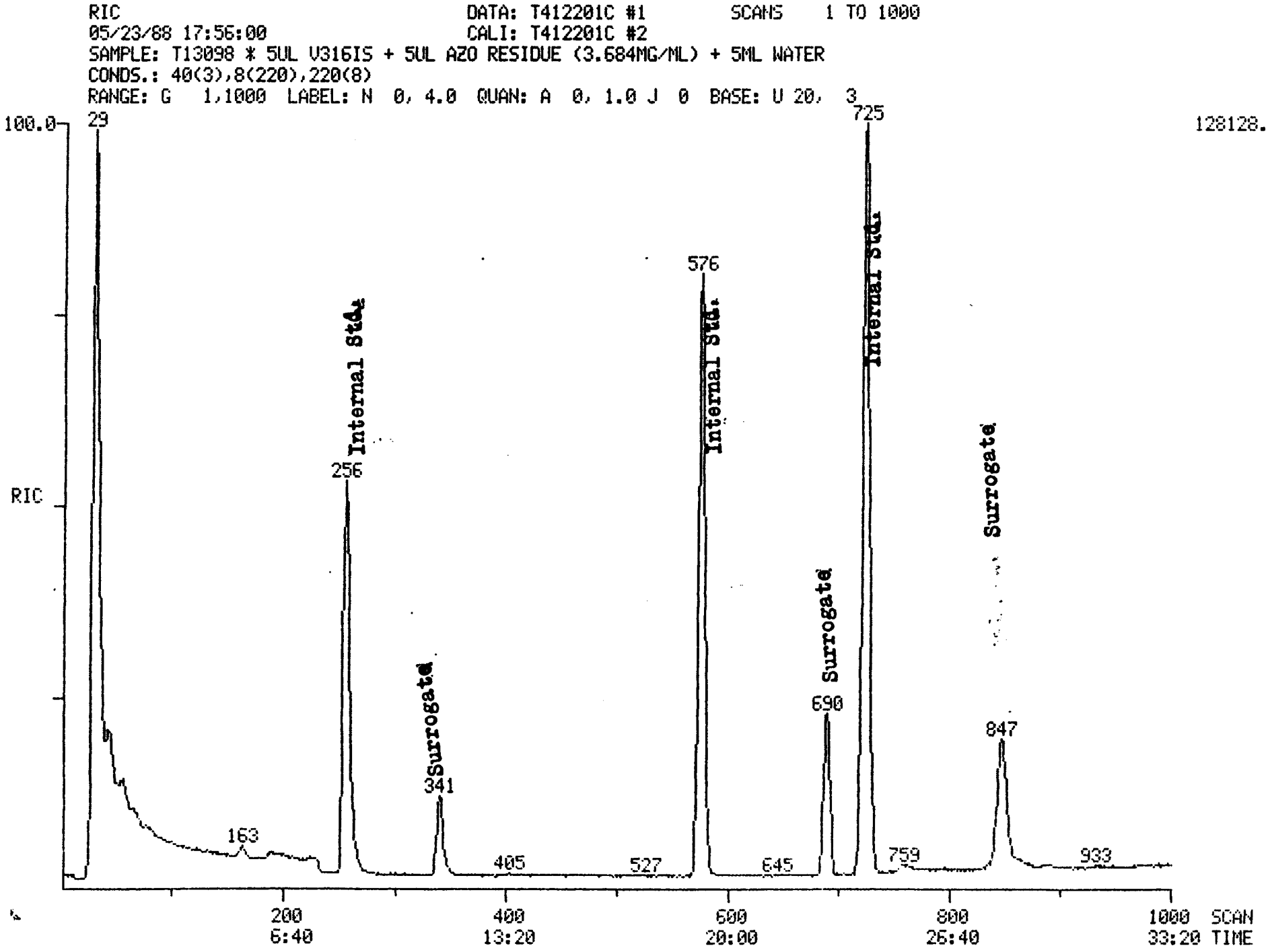
Client: TERRAN ST Date: 5/23/88 & 5/24/88

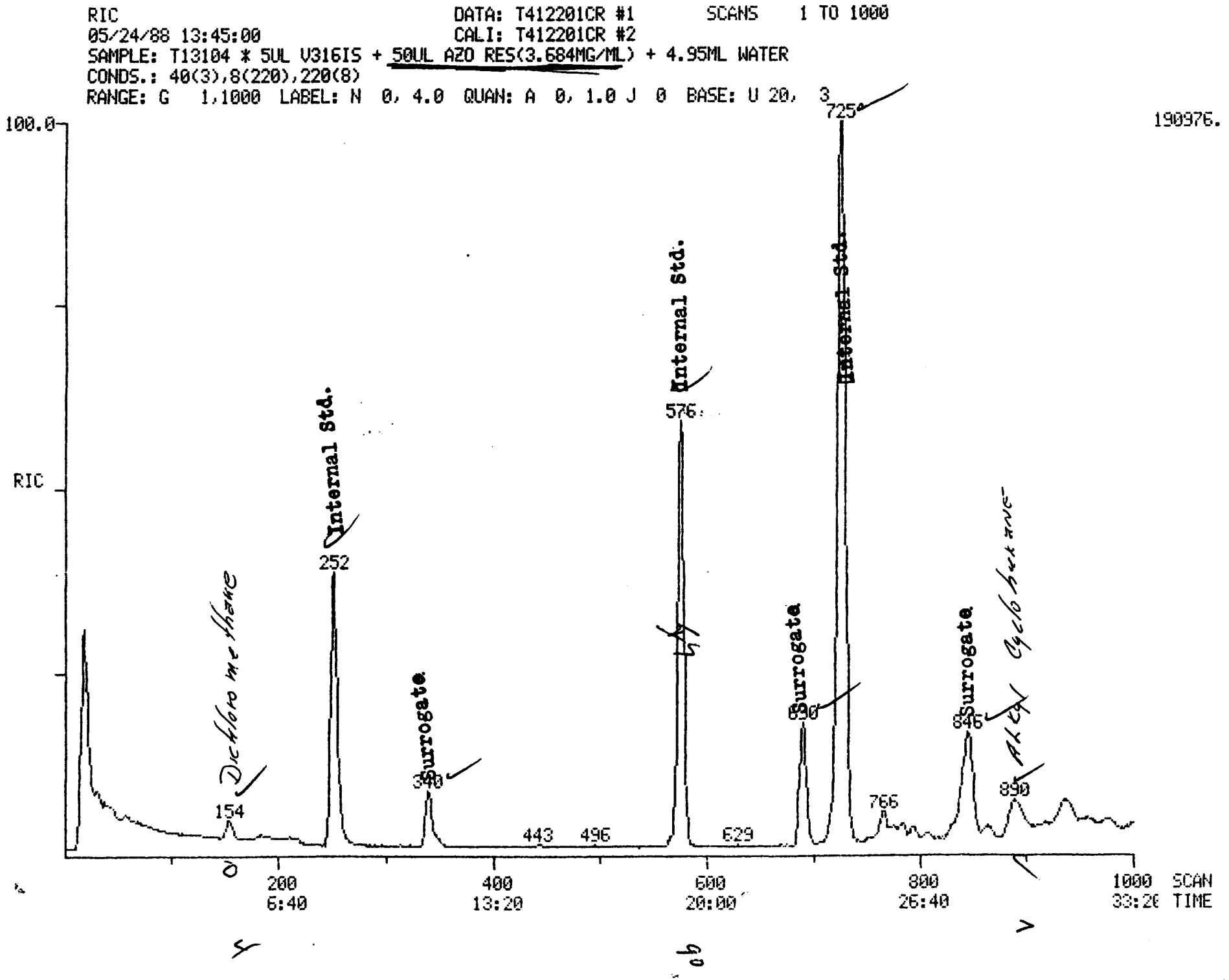
PN: 4874 Analyst: D. Melton

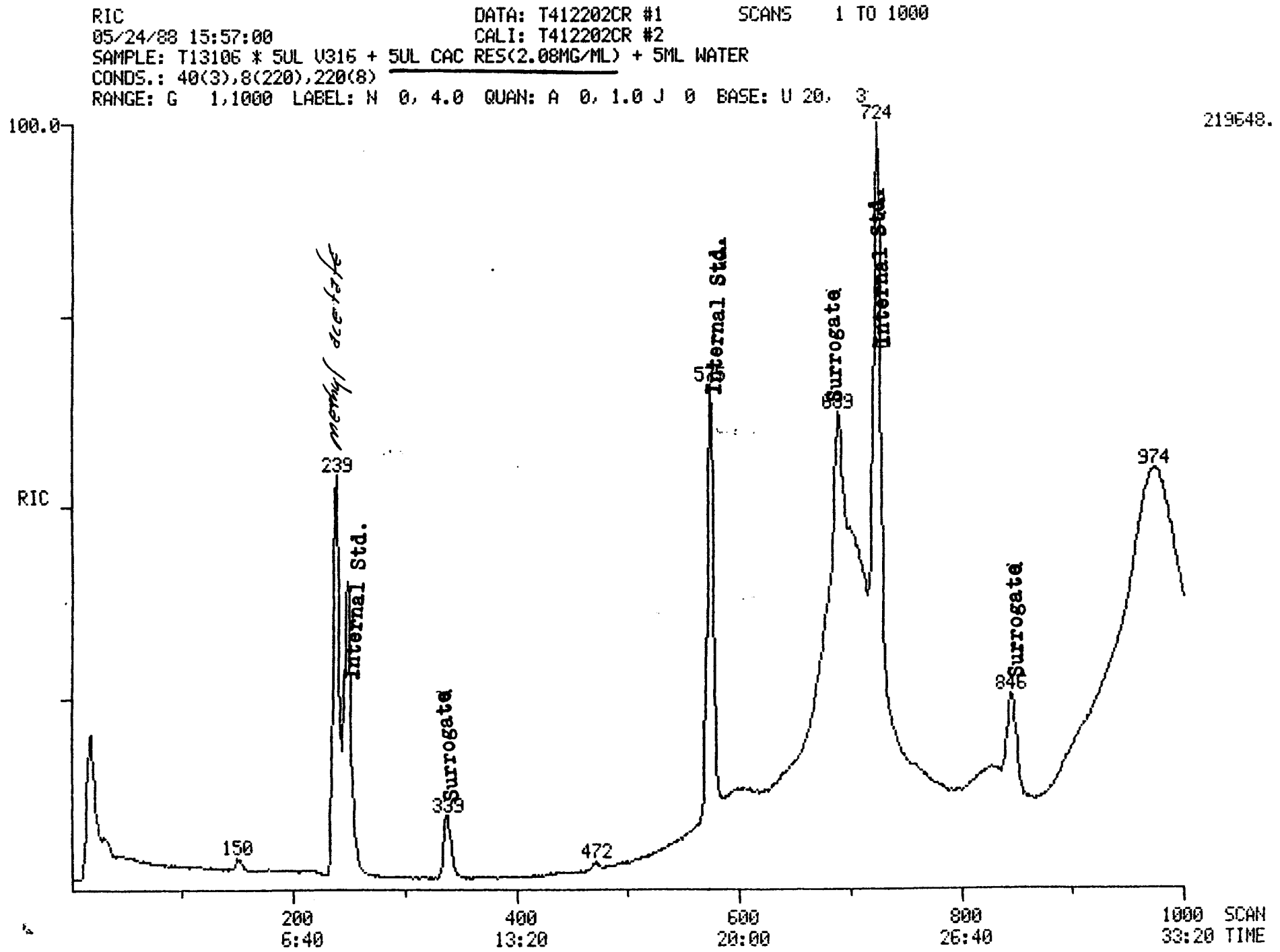
Work Order: T8-04-122

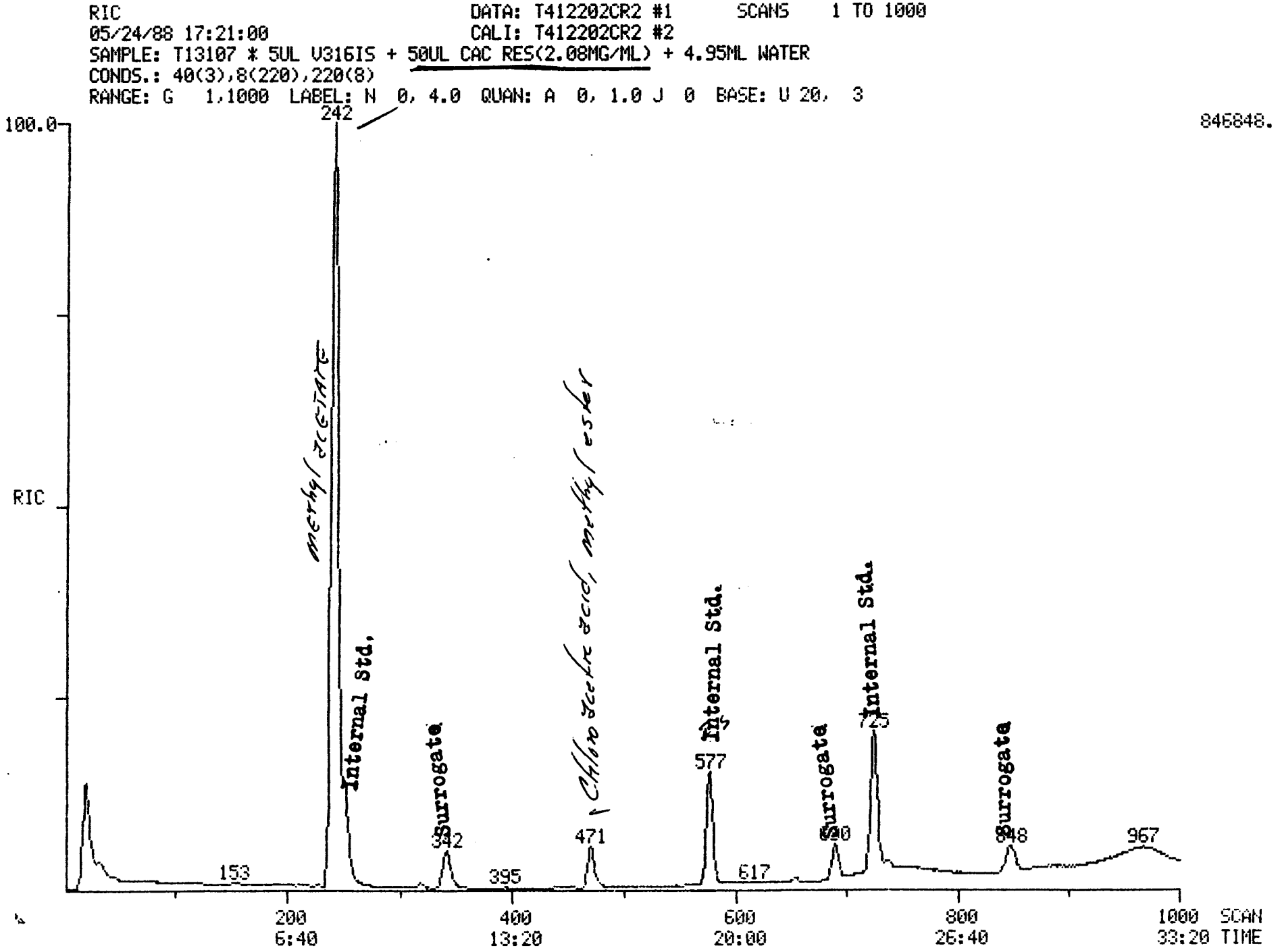
Matrix: Residue Samples

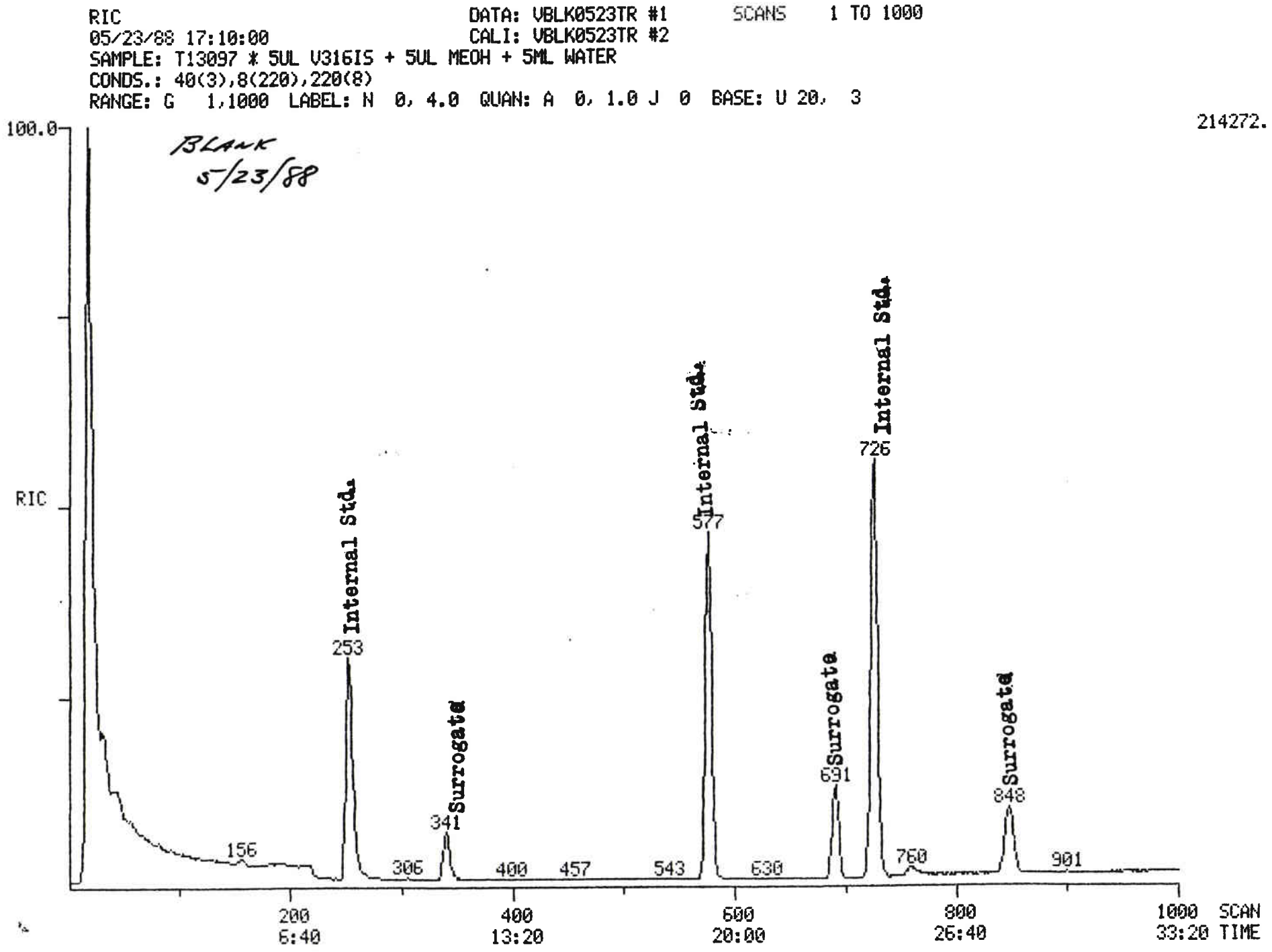
Sample ID	PEI No.	Toluene-d8	BFB	1,2-Dichloroethane-d4
Blank	VBLK0523T	100	102	98
AZO Resin MeOH	T4122O1C	100	100	98
CAC Resin H2O	T4122O2C	100	99	96
Blank	VBLK0524T	100	101	61
AZO Resin MeOH	T4122O1CR	97	103	61
CAC Resin MeOH	T4122O2CR	114	102	61
" " " "	T4122O2CR2	109	102	63

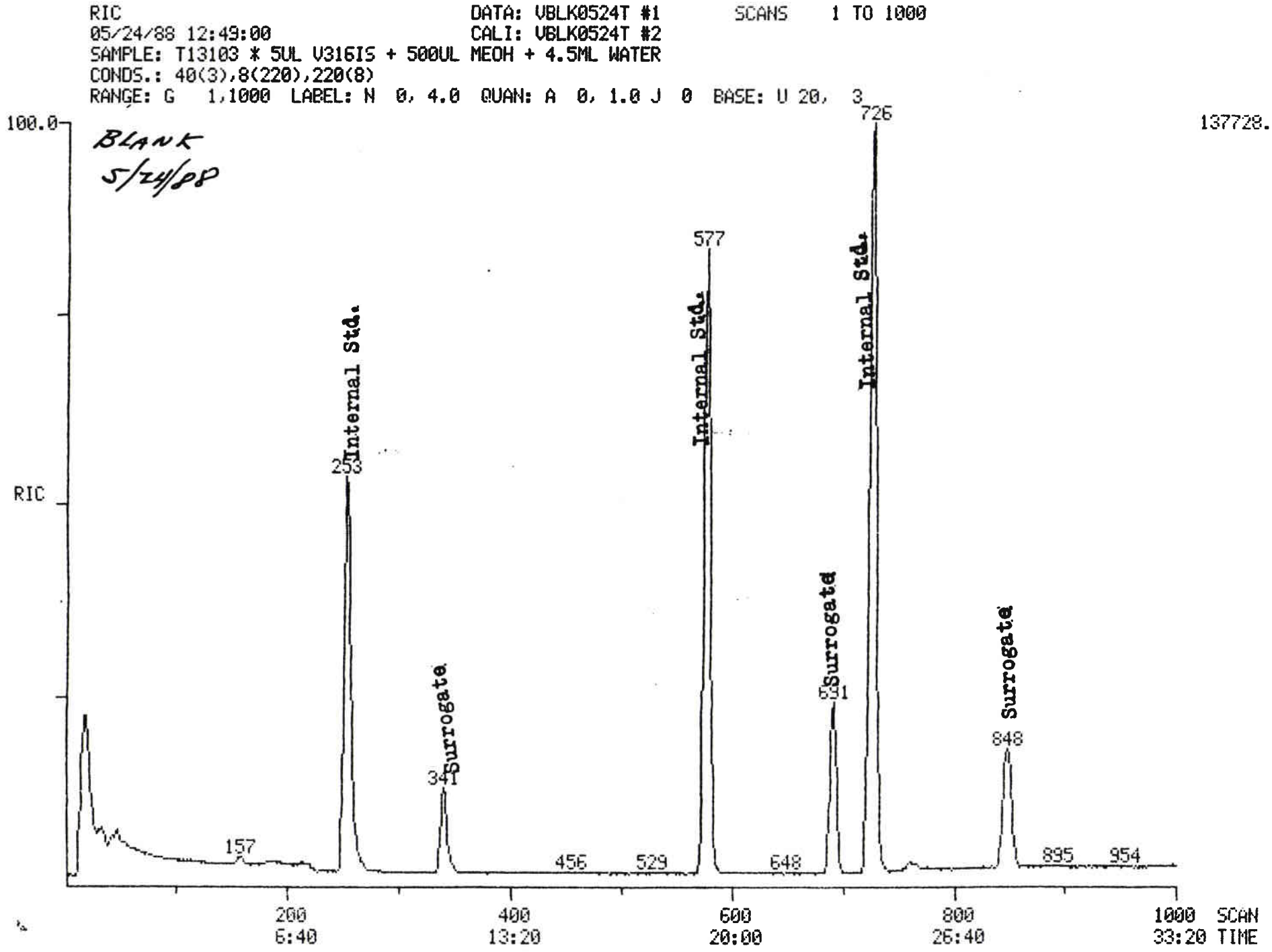

RIC
DATA: T412201C #1
SCANS 1 TO 1000
05/23/88 17:56:00
CALI: T412201C #2
SAMPLE: T13098 * 5UL V316IS + 5UL AZO RESIDUE (3.684MG/ML) + 5ML WATER
CONDS.: 40(3),8(220),220(8)
RANGE: G 1,1000 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3
100.0
128128.
RIC
29
256
Internal Std.
341
Surrogate
163
405
527
576
Internal Std.
645
690
Surrogate
725
Internal Std.
759
847
Surrogate
933
200
6:40
400
13:20
600
20:00
800
26:40
1000 SCAN
33:20 TIME


RIC
DATA: T412201CR #1
SCANS 1 TO 1000
05/24/88 13:45:00
CALI: T412201CR #2
SAMPLE: T13104 * 5UL V316IS + 50UL AZO RES(3.684MG/ML) + 4.95ML WATER
CONDS.: 40(3),8(220),220(8)
RANGE: G 1,1000 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3
100.0
190976.
RIC
Internal Std.
252
Surrogate
Internal Std.
576
Surrogate
Internal Std.
725
Surrogate
154
443
496
629
766
200
6:40
400
13:20
600
20:00
800
26:40
1000 SCAN
TIME


RIC
05/24/88 15:57:00
DATA: T412202CR #1
CALI: T412202CR #2
SCANS 1 TO 1000
SAMPLE: T13106 * 5UL V316 + 5UL CAC RES(2.08MG/ML) + 5ML WATER
CONDS.: 40(3),8(220),220(8)
RANGE: G 1,1000 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3
100.0
219648.
RIC
239
Internal Std.
333
Surrogate
150
472
Internal Std.
724
Surrogate
846
974
200
6:40
400
13:20
600
20:00
800
26:40
1000 SCAN
33:20 TIME


RIC
DATA: T412202CR2 #1 SCANS 1 TO 1000
05/24/88 17:21:00 CALI: T412202CR2 #2
SAMPLE: T13107 * 5UL V316IS + 50UL CAC RES(2.08MG/ML) + 4.95ML WATER
CONDS.: 40(3),8(220),220(8)
RANGE: G 1,1000 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3
100.0
846848.
RIC
242
153
Internal Std.
Surrogate
342
395
471
577
617
Internal Std.
Surrogate
720
Internal Std.
25
Surrogate
848
967
200
6:40
400
13:20
600
20:00
800
26:40
1000 SCAN
33:20 TIME


RIC
DATA: VBLK0523TR #1
SCANS 1 TO 1000
05/23/88 17:10:00
CALI: VBLK0523TR #2
SAMPLE: T13097 * 5UL V316IS + 5UL MEOH + 5ML WATER
CONDS.: 40(3),8(220),220(8)
RANGE: G 1,1000 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3
214272.
100.0
BLANK
5/23/88
RIC
Internal Std.
253
Surrogate
341
156
306
400
457
543
Internal Std.
577
630
Surrogate
691
Internal Std.
726
760
Surrogate
848
901
200
6:40
400
13:20
600
20:00
800
26:40
1000 SCAN
33:20 TIME


RIC
05/24/88 12:49:00
DATA: VBLK0524T #1
CALI: VBLK0524T #2
SCANS 1 TO 1000
SAMPLE: T13103 * 5UL V316IS + 500UL MEOH + 4.5ML WATER
CONDS.: 40(3),8(220),220(8)
RANGE: G 1,1000 LABEL: N 0, 4.0 QUAN: A 0, 1.0 J 0 BASE: U 20, 3
100.0
137728.
BLANK
5/24/88
RIC
Internal Std.
Surrogate
Internal Std.
Surrogate
Internal Std.
Surrogate
253
34
577
55
726
848
157
456
529
648
895
954
200
6:40
400
13:20
600
20:00
800
26:40
1000 SCAN
33:20 TIME

LABORATORY DATA

Client Terran ST
PN 4874 Date
Analyst

Analysis Summary
Method Number
Checker

T8-04-122

	AZO residue sample 01	CAC residue sample 02				
Flashpoint						
Viscosity						
CN						
S=						
H_2O Act						
As ug/g	0.26	<0.20				
Cd	<0.07	<0.07				
Cr	0.40	0.27				
Pb	<6.2	<6.2				

TABLE VI-2. SUMMARY OF ANALYSIS METHODS, CAC INCINERATOR TRIAL BURN, MONSANTO-QUEENY PLANT, ST. LOUIS, MISSOURI (Continued)

Sample	Analytical parameters	Preparation method	Analysis method
	Moisture	N/A	ASTM D95 xylene codistill
	Ash	N/A	ASTM D482 Ignition
	Organochlorine	EPA 3580 dilution	EPA 9020 Microcoulometry
	Viscosity	N/A	ASTM D445 viscometer
Utility water	POHCs	EPA 5030 purge&trap	EPA 8240 GC/MS
	Total chloride	N/A	EPA 300.0 Ion chromatography
	TOX	N/A	EPA 9020 Microcoulometry
	Suspended solids	N/A	EPA 160.1 filter, filtrate evaporation, & gravimetric
	Dissolved solids	N/A	EPA 160.2 filter, residue desiccation, & gravimetric
Quench effluent	POHCs	EPA 5030 purge&trap	EPA 8240 GC/MS
	Total chloride	N/A	EPA 300.0 Ion chromatography

TABLE VI-2. SUMMARY OF ANALYSIS METHODS, CAC INCINERATOR TRIAL BURN, MONSANTO-QUEENY PLANT, ST. LOUIS, MISSOURI (Continued)

Sample	Analytical parameters	Preparation method	Analysis method
	TOX	N/A	EPA 9020 Microcoulometry
	Suspended solids	N/A	EPA 160.1 filter, filtrate evaporation, & gravimetric
	Dissolved solids	N/A	EPA 160.2 filter, residue desiccation, & gravimetric
Scrubber effluent	POHCs	EPA 5030 purge&trap	EPA 8240 GC/MS
	Total chloride	N/A	EPA 300.0 Ion chromatography
	TOX	N/A	EPA 9020 Microcoulometry
	Suspended solids	N/A	EPA 160.1 filter, filtrate evaporation, & gravimetric
	Dissolved solids	N/A	EPA 160.2 filter, residue desiccation, & gravimetric
Stack gas	POHCs/PICs	EPA 5040 Thermal desorb	EPA 8240 GC/MS
Stack gas	POHC/PICs	EPA 3510 Separatory funnel	EPA 8270 GC/MS
		EPA 3540 Soxhlet extraction	EPA 8270 GC/MS